# PROGRESS REPORT ON CONVENTIONAL STACK SAMPLING AT ST. LAWRENCE CEMENT



Ministry of the Environment
Ontario

The Honourable
Keith C. Norton, Q.C.,
Minister

Gérard J. M. Raymond
Deputy Minister

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# PROGRESS REPORT ON CONVENTIONAL STACK SAMPLING AT ST. LAWRENCE CEMENT

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Prepared By:

Air Resources Branch

Ministry of the Environment

880 Bay St., 4th Fl.,

Toronto, Ontario.

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TABLE OF CONTENTS

## 1.0 INTRODUCTION:

### 1.1 Objective of Conventional Sampling During Burn:

A conventional sampling method for determination of PCBs in the exhaust gases will be used for measuring of the emissions of PCBs from the St. Lawrence Cement Kiln No. 1 during the experimental burn of waste oils containing PCBs. The collected samples will also be analysed for other organic materials that may be emitted from the kiln along with PCBs, thus complementing and at the same time providing an independent check on the measurements of PCBs obtained by the TAGA 3000.

### 1.2 Preliminary Conventional Sampling and Its Objectives:

Ideally, any measurement method when applied to a "new source", should be capable of producing valid results, regardless of conditions at that source. However, as it has been repeatedly shown in practice, almost every source has its own idiosyncracies and presents a new challenge to the measuring crew. In order to circumvent the peculiarities specific to the source in question and do valid measurements in a short time, such as during the experimental burn at the St. Lawrence Cement plant, the source should be examined beforehand and the method adjusted as necessary.

Information of this kind was generated in previous sampling programmes at the St. Lawrence Cement Kiln #1. (Reference 1,2). However, because of new developments in sampling and analytical methods (reference 3), involvement of different personnel in the present study, additional sampling of the clinker cooler exhaust gases and possibly different chemistry of the precipitator kiln exhaust gases it was concluded that more sampling when PCBs are not deliberately fed into the kiln should be performed. The objective of this sampling would be to measure the background levels of PCBs, background levels of organic impurities

that can interfere with analysis and to check the extraction and analytical procedures.

This work was done at the St. Lawrence Cement plant on two occasions and this report summarizes activities and the results obtained so far. However, it should be pointed out that the collected samples are undergoing secondary analyses in the laboratories and that this report is not complete by any means. It is rather a progress report presenting the results obtained to date.

## 2.0 SUMMARY:

Two sampling programmes, described in detail in the next item of this report were carried out so far. The results suggest that PCBs may have been present in the stack gases of the Cement Kiln #1 when no PCBs were knowingly and deliberately added into the kiln. The concentrations ranged from .88 micrograms per cubic metre of stack gases to 2.45 micrograms per cubic metre of stack gases.

However, on the basis of the analytical results generated so far it cannot be stated with certainty that these were indeed PCBs. Some other organic compounds may have interfered with the analyses and consequently could have been erroneously interpreted as PCBs. Work on verification of the results of chemical analysis is presently still in progress. On the other hand, if these compounds are proven to be PCBs, their levels are still low and far below the targeted level of 20 micrograms per cubic meter of stack gas, the latter being the safe level as indicated in reference 5.

Regardless of whether or not these compounds are PCBs they must be treated as background impurities present in the stack gases when no PCBs are burned in the kiln. They affect the sensitivity of the method of determination of PCBs and limit this sensitivity to the levels at which they are present in the stack gas. From the measurements carried out so far it can be stated that this sensitivity is adequate to detect any "new" PCBs in the stack gas at the levels above approximately 2.5 micrograms per cubic metre, which again is far below

the safe levels of 20 micrograms per cubic meter of stack gas.

In order to learn more about the variation of these background impurities when no PCBs are burned in the kiln, more sampling is planned for the near future. It will then be possible to further define the sensitivity of the sampling method and statistically characterize its variation.

## 3.0 DESCRIPTION OF ACTIVITIES:

Stack sampling crews of Envirocon (Eastern) Limited performed complete sampling of emissions from Kiln No. 1 on November 1, 1979. Gases carrying particulates and other constituents of the emissions were sampled from the clinker cooler stack and the kiln precipitator exhaust duct. An interim summary report prepared by Envirocon (Eastern) Limited describing these activities can be found in Appendix 1. Activities described in the original work description issued by the Ministry of the Environment (Reference 4) and based on EPA Method 5 were modified, in that two sampling trains were used in sampling of the precipitator exhaust gases in this first sampling campaign.

After the sampling was completed both trains were brought to the Ministry's laboratories at 880 Bay Street where the samples from one train were modified ("spiked") with small known quantities of several PCBs isomers. The trains were then sent to Nucro-Technics Limited for sample recovery and extraction. (Nucro Technics Limited have been subcontracted by Envirocon (Eastern) to perform all cleaning of glassware of sampling equipment and extractions of samples)and a report prepared by Nucro-Technics Limited describing their procedures is attached as Appendix 2.

After extraction, the samples were then transported to the Pesticide Laboratory of the Ministry's Laboratory Services Branch for final processing

and analyses. The laboratory procedures on the sample extracts are described in the report by the Pesticide Laboratory in Appendix 3. Part of the extracts of some samples were also analysed by the Monitoring and Instrumentation Development Unit of the Ministry and their report can be found in Appendix D.

The same activities were repeated on January 3, 1980, except that, in this test, only the kiln precipitator exhaust gases were sampled. Background levels of PCBs in the first test at the clinker cooler stack appeared low and because of that it was concluded that there was no need to repeat this sampling.

As described in Appendices 3 and 4 three different analytical methods were used in the analyses of samples; two gas chromatographic methods, (one utilizing a glass capillary column, another a packed column) and a prechlorination technique. Selected samples are also being analysed by a gas chromatographic and mass spectrometric technique to further verify the PCB analyses obtained by other techniques.

## 4.0 DISCUSSION:

Stack sampling tests on November 1, 1979 were described in detail in Appendix 1. The samples from the train designated as train #2 were "spiked" with known quantities of a PCB mixture of known composition. These "spike" quantities are given in Appendix 4. After extraction and analysis of samples it became obvious that background PCBs were present in the samples in much larger quantities than the "spikes", and that these large quantities may have prevented exact determination of the added PCBs. Also the determination and identification of PCBs in the samples has been further complicated with the fact that a very complex mixture of organic material has been identified. This somewhat unexpected discovery had given rise to speculations that these impurities were possibly released from the sampling train and more specifically from the teflon and fiberglass tapes used to seal the filter holders. However, such a simple explanation was recognized as possibly erroneous because the samples from the clinker cooler stack, where a train "identical" to those at the kiln precipitator exhaust was used, were relatively "clean".

It was therefore, decided to repeat sampling at the kiln precipitator duct with the trains modified in that no teflon or fiberglass tapes were to be used for sealing of the filter holder. This sampling was done on January 3, 1980.

The amount of "spikes" on the samples was this time approximately double in comparison with earlier samples; however, the results were still high and difficult to interpret. It became obvious that the kiln precipitator exhaust gases contain organic materials and possibly PCBs in higher than expected levels. As indicated in the discussion part of the reports by the Pesticide Laboratory and the Monitoring and Instrumentation Development Unit a positive identification of PCBs in any of the samples collected so far was not yet made. Whether or not the organic materials, expressed as PCBs, in quantities shown in Appendices 3 and 4, are the true PCBs they should still be interpreted as background impurities. Since their level can change, depending on the composition of the raw materials fed into the kiln and on the process conditions, it became obvious that a more detailed examination will have to be carried out in the near future. It is anticipated that additional 5 to 10 sampling campaigns similar to those described in this report will be sufficient for this purpose.

## 5.0 CONCLUSIONS AND COURSE OF FURTHER ACTION:

1) The background impurities, analysed but still not positively identified as PCBs, were measured in the kiln precipitator exhaust gases at levels above 2 micrograms per cubic metre of stack gas when no PCBs were burned in the kiln.

2) Sensitivity of the conventional stack sampling method was determined to be 2.5 micrograms of PCBs in one cubic metre of stack gas.

3) Additional sampling of the kiln exhaust gases when no PCBs are burned in the kiln is needed to define the background level of organic impurities.

References


1. Burning Waste Chlorinated Hydrocarbons in a Cement Kiln, Report EPS 4-WP-77-2, Fisheries and Environment Canada, March 1977.

2. Sampling and Analysis of Emissions from a Wet Process Kiln at St. Lawrence Cement Company in Mississauga, (ORF Report No. P-2818/G (Revised)-02), November 27, 1978.

3. EPA Draft Method for Determination of PCBs in Industrial, Sewage Sludge and Municipal Refuse Incinerators, February, 1978.

4. Request for Proposal for Stack Sampling for Chlorinated Compounds at the St. Lawrence/Mississauga Cement Plant, Air Resources Branch, July 12, 1979.

5. Proposed Protocol for the Incineration of Polychlorinated Biphenyls at St. Lawrence Cement Ltd., Mississauga, Air Resources Branch, 1979.

# INTERIM SUMMARY REPORT
# ONTARIO MINISTRY OF THE ENVIRONMENT
# PCB INCINERATION STUDY

Prepared by:

Envirocon (Eastern) Ltd.
P.O. Box 1339
Station "B"
Downsview, Ontario
M3H 5W3

Telephone (416) 661-1350

TE 2942

January 1980

## SUMMARY

This summary report details activities to date with regard to Ministry of the Environment Purchase Order Number A61658.

This contract was awarded to Envirocon (Eastern) Ltd. and involves the source emissions measurement component of the PCB study being conducted at the St. Lawrence Cement Plant at Clarkson, Ontario.

The report summarizes all activities up to and including the two background source test surveys, and includes the Laboratory Analysis Procedures. The sample analysis portion of this study was conducted by Nucro-Technics Laboratory Limited on behalf of Envirocon (Eastern) Ltd.

## 1.0 INTRODUCTION

Envirocon (Eastern) Ltd. was retained by the Ontario Ministry of the Environment to conduct the source measurement component of the PCB study being conducted at the St. Lawrence Cement Plant in Clarkson, Ontario.

This interim, summary report details activities up to and including the two on-site background source measurement tests and includes the following:

Field Preparation and Sample Collection:

1) Equipment utilized
2) Back up, proofing and check procedures
3) Sampling Procedures
4) Preliminary field data

Laboratory Analysis Procedures:

1) Analytical Equipment utilized
2) Sample handling
3) Procedures
4) Preliminary analytical results

## 2.0 EQUIPMENT UTILIZED

Five sample trains were used in the November 1 test and four were used in the January 3 test.

The sample trains used were Isotrains manufactured by Envirocon (Eastern) Ltd. in accordance with provincial, federal and EPA testing code standards. Figure 1 is a schematic of the equipment. The Isotrain is of heavy duty construction, capable of maintaining the 400°F temperatures in the probe and sample box required during this survey.

To meet the specific testing requirements of this survey, modifications had to be made in the Isotrain:

(i) The filter holder had to accomodate a glass frit
(ii) Glass probe liners were required
(iii) A florisil adsorbent cartridge was required in the impinger section of the train.
(iv) Silicone grease could not be used as a vacuum sealant.

Incorporation of a glass frit in the filter holder caused serious leakage problems for the November 1 tests. To stop air leaking through the sides of the frit, the sides were wrapped in teflon tape. The filter holder was then held together by high temperature glass fibre tape in lieu of the standard filter holder. The frits and filter holders were modified for the January 3 test so that the frits fit right inside the filter holder thereby, eliminating the leakage problem.

Teflon caps were provided by Ace Glass Co. to be used as gaskets for the ground glass joints, in place of using silicone grease. The teflon caps were too hard and did not fit well enough to provide a good seal. After experimentation, this problem was overcome by using soft teflon tape as the gasketing material on the ground glass surfaces.

## GAS CONTACTING COMPONENTS

Each sample train was composed of the following items arranged in order from the nozzle to the silica gel:

nozzle, heated glass probe liner, cyclone by-pass, front filter holder, filter, frit, rear filter holder, filter to impinger connector, 2 impingers each containing 100 mls of PCB free water, 3 empty impingers, (U tubes connecting the previous five impingers), impinger to cartridge piece, cartridge containing Florisil, cartridge to impinger piece, impinger containing silica gel.

All the above pieces are made of pyrex with the exception of the nickel plated nozzle.

## 3.0 SURVEY PROCEDURES

### 3.1 Flow Measurement

Velocity measurements were taken using a pre-calibrated S-type pitot tube in conjunction with an inclined manometer or a differential pressure Magnehelics Gauge. The equal area method, as specified by the Source Testing Code, was followed to determine the volumetric flow rate. The stack was divided into a number of zones of equal area. The velocities were determined within each zone, summed and averaged. Temperature, stack static pressure, and atmospheric pressure were recorded and molecular weight and moisture content determined to enable correction of flow to dry standard conditions of $70^{o}F$ and 29.92" Hg.

### 3.2 Stack Temperature Measurements

Temperature measurements were made simultaneously with the velocity measurements. A commercial chromel-alumel Type K thermocouple in conjunction with a Thermo-electric ELPH3 digital temperature indicator was used to measure stack temperatures. The temperature indicators were calibrated beforehand with a certified calibrated potentiometer.

### 3.3 Stack Gas Composition

Exhaust stack gas composition was determined by conducting Orsat analysis for $O_2$, $CO_2$ and CO during the testing.

## 3.4 Particulate Sampling

Particulate loadings were obtained using an approved sampling train as specified in the Source Testing Code. A schematic of the sampling train is shown in Figure 1. Particulate sampling methodology was in accordance with the Ontario Ministry of the Environment Source Testing Code, and is detailed below.

### 3.4.1 Particulate Sampling Methodology

(a) Preliminary velocity, temperature and moisture characteristics were determined.

(b) Isokinetic sampling rates and nozzle sizes were calculated from preliminary data. Nozzle size was selected and sampling duration was set to allow collection of an acceptably large sample volume.

(C) Sampling units were vacuum checked in accordance with the Source Testing Code.

(d) Traversing was conducted by the standard method of dividing the cross sectional stack into equal area zones and sampling at the centroid of each zone for a prespecified time per point. Sampling rates were adjusted as necessary to maintain a sampling rate within the specified limits of isokinetic deviation (100 $\pm$ 10 per cent) on a point by point basis. Fiberglass filter paper, with a 0.3 u DOP efficiency of 99.7%, was used to collect the particulate matter.

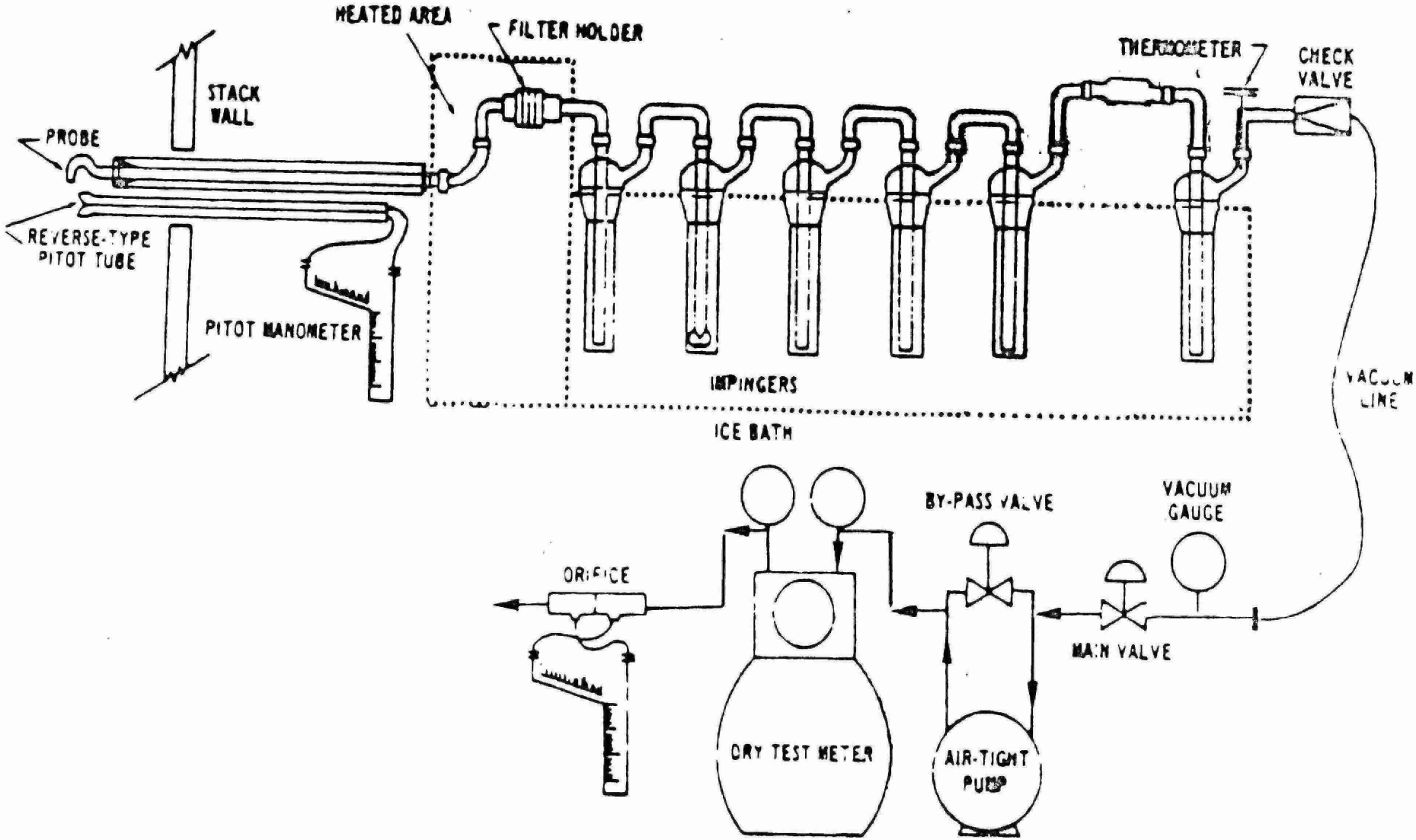


FIGURE 1 PARTICULATE SAMPLING TRAIN

## 3.5 Sampling Procedure

### 3.5.1 Test #1, (November 1, 1979)

#### 3.5.1.1 Kiln Exhaust

Five sample trains were cleaned at Nucro Technics to a proofing level of less than 50 nanograms detectable PCBs. Glass pieces were sealed in cleaned aluminum foil and transported to Envirocon Eastern's facility where the trains were completely assembled and leak checked. The open ends of the sampling train were covered with cleaned foil and the trains were transported to the test site.

Two simultaneous isokinetic tests were performed on the cement kiln exhaust, in accordance with the Source Testing Code.

Each test included five traverses, with eight points per traverse, at five minutes per point. The total sampling time was 360 minutes and the total volume extracted was 120 $ft^3$ per train. Leak checks were performed before each traverse and after the final traverse. The volume drawn for each leak check was recorded.

#### 3.5.1.2 Clinker Exhaust

In addition to the tests on the kiln exhaust, a simultaneous isokinetic test was performed on the clinker cooler.

Measurements were taken at the 72" ID exhaust stack at two sampling ports located at right angles to each other on the same horizontal plane.

The test included two traverses, with 18 sampling points per traverse,

4 readings per point at 2.5 minutes per reading for a total sampling time of 360 minutes.

The probes were heated and the temperature of the hot boxes was maintained above 250°F (usually at about 375°F) throughout the test.

After completion of the simultaneous tests, a third train with a heated probe and a hot box maintained at 375°F, was used at the plant to draw a volume of ambient air approximately equal to the total volume drawn in the leak checks of each train. The trains were sealed with clean foil and, still assembled, taken to MOE for spiking, then to Nucro Technics for disassembly, washup and analysis.

### 3.5.2 Test #2, (January 3, 1980)

Four sample trains were cleaned at Nucro Technics and verified to 50ng. PCBs. The trains were completely assembled and leak checked at Envirocon (Eastern) Ltd., the open ends were covered with cleaned foil and the trains were transported to the test site.

Two simultaneous isokinetic tests were performed in accordance with the Source Testing Code.

Each test included five traverses, with eight points per traverse, at five minutes per point. The total sampling time was 200 minutes and the total volume extracted was 100 $ft^3$ per train. Leak checks were performed before each traverse and after the final traverse. The volume drawn for each leak check was recorded.

The probe was heated and the temperature of the hot box was maintained above 250°F (usually at about 375°F) throughout the test.

After completion of the simultaneous tests a third train, with a heated

probe and a hot box maintained at 375°F, was used at the plant, to draw a volume of ambient air approximately equal to the total volume drawn in the leak checks of each train. The trains were sealed with clean foil and returned, still assembled, to MOE then to Nucro Technics, for analysis.

### 3.5.3 Background Tests

In addition to the on-site tests, two lab tests were performed to provide a background PCB measurement.

The test consisted of pulling 100 $ft^3$ of air through a train at the same average rate as the on-site tests with the train maintained at the same approximate temperature.

Another Florisil cartridge was connected in front of the nozzle to remove any ambient PCBs. The trains were cleaned and analysed in the same manner as the trains used in the field source tests.

## 4.0 BACKUP, PROOFING AND CHECK PROCEDURES

The following steps were taken to insure the reliability of the results.

1) Instrument calibrations were conducted on:
   (a) gas meter - Bell Prover system - Rockwell
   (b) pitots (before and after tests) - University of Toronto mechanical engineering wind tunnel
   (c) Magnehelics - inclined manometer
   (d) Temperature indicators - reverse potentiometry

2) All gas contacting train components were cleaned before assembly and verified to be less than 50 ng PCB. Analysis results are attached.

3) All trains were assembled and leak checked in the clean laboratory of Envirocon (Eastern) Ltd.

4) The inlet and outlet of the trains were sealed with PCB free aluminum foil.

5) The trains were transported intact from Envirocon (Eastern) Ltd., to St. Lawrence Cement, MOE (Bay St.) and Nucro Technics.

6) A spare train was on-site for both tests in case of breakdown or breakage.

7) Duplicate simultaneous runs on the kiln exhaust were performed. At the beginning of each run and at the completion of each traverse, vacuum leak checks were conducted to ensure a leakage rate of less than .02 $ft^3$/min at 2" Hg. higher than the maximum vacuum encountered during the testing.

8) Leak checks conducted on the train were compensated by drawing equivalent volumes of ambient air through a blank train located on-site.

9) Samples were spiked by MOE as a check on the analysis.

## 5.0 RESULTS - PROBLEMS ENCOUNTERED

The only problems encountered during the actual sampling are outlined below. Corrective action was taken so as to minimize error through leakage or contamination.

A leak in excess of 0.02 $ft^3$/min and a torn filter were observed during the leak check following the first traverse of the January 3, 1980 test. The filter holder was tightened and the test has continued with a torn filter.

A cyclone by-pass was broken at the same time and had to be replaced by a clean spare one.

The probe liner cracked during the first background laboratory test and did not leak check, therefore, the test was discarded. No problems were encountered during the second laboratory test.

# APPENDIX 2

NUCRO TECHNICS LIMITED

ANALYTICAL REPORT

2000 ELLESMERE ROAD, UNIT 16, SCARBOROUGH, ONTARIO M1H 2W4 416/438-6727

DATE January 15, 1980

PROJECT NO. 24769B

Mr. John Trought, P. Eng.
Manager
Envirocon (Eastern) Limited
P.O. Box 1339
Downsview, Ontario
M3H 5W3.

SUBJECT CLEANING & PROVING OF GLASS SAMPLE CONTAINERS PROVIDED BY MINISTRY OF THE ENVIRONMENT

A) Procedures

Cleaning of 8 oz. and 32 oz. wide mouth glass sample containers for process samples and extracts. Glass containers were cleaned internally by solvent rinsing with glass distilled solvents. The following sequence of solvent rinsings was employed:

| | |
|---|---|
| Methylene chloride | 1X |
| Acetone | 2X |
| Hexane | 2X |

A final rinse with pentane was discarded. A subsequent rinse with pentane of the interior surfaces was carried out and collected in a 600 milliliter Kuderna-Danish concentrator. Containers were cleaned and proven in batches of 12 pieces.

A keeper of 0.5 milliliter iso-octane and two or three precleaned boiling chips were added.

Samples were concentrated to approximately 5 milliliters employing the main body of the Kuderna-Danish concentrator and a three ball macro Snyder column on a water bath at 60°C.

The main body of the Kuderna-Danish was removed and a macro Snyder column substituted for the macro Snyder column. The concentration was continued on a heating apparatus to 0.5 milliliters or less, then made up to 0.5 milliliters.

The iso-octane concentrate was transferred to precleaned glass micro vials with aluminum foil lined caps for holding until GLC assay.

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SUBJECT CLEANING & PROVING OF GLASS SAMPLE CONTAINERS PROVIDED BY MINISTRY OF THE ENVIRONMENT

DATE January 15, 1980.

PROJECT NO. 24769B

Results of GLC Assay - See Table 1.

Table 1 - Results of Proving Assay for glass containers for Process Samples

| Designation | Concentration PCB ng/ml | Total PCB in ng for the Batch of 12 pieces |
|---|---|---|
| 1 - 12 - 8 | 82.9 | $\leq$ 41.5 |
| 2 - 12 - 8 | $\leq$ 48.77 | $\leq$ 24.4 |
| 3 - 12 - 8 | $\leq$ 3.45 | $\leq$ 1.8 |
| 4 - 12 - 8 | $\leq$ 37.8 | $\leq$ 18.9 |
| 5 - 12 - 8 | $\leq$ 16.9 | $\leq$ 8.5 |
| 6 - 12 - 32 | $\leq$ 31.8 | $\leq$ 15.9 |

B) A number of supplementary batches of 32 oz. containers were cleaned and proven for use in the storage of stack samples and process samples extracts. A more extensive cleaning procedure was employed.

The glass containers were soaked in Decon 75 - Water solution overnight and rinsed thoroughly with tap water followed by distilled water. The glass containers were then solvent rinsed with glass distilled solvents. The following sequence of solvent rinsings was employed:

| | |
|---|---|
| Methylene chloride | 1X |
| Acetone | 2X |
| Hexane | 2X |

After air drying the containers were baked out for a minimum of 12 hours in a 250°C forced air oven. After cooling the containers received a pentane rinse to waste. A final pentane rinse of the internal surfaces was collected in a Kuderna-Danish concentrator. Containers were cleaned and proven in batches of 12 pieces.

Concentration and assay were carried out as previously reported.

Table 2 - Results of Proving Assay for Glass Containers for Extracts.

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SUBJECT CLEANING & PROVING OF GLASS SAMPLE CONTAINERS PROVIDED BY MINISTRY OF THE ENVIRONMENT

DATE January 15, 1980

PROJECT NO. 24769B

| Designation | Concentration PCB ng/ml | Total PCB in ng for a batch of 12 pieces |
|---|---|---|
| Batch 1 | 63.24 | 31.6 |
| Batch 2 | 6.10 | 3.1 |

Results of GLC Assay - See Table 2

Fred S. Seymour, B.Sc.
Director of Technical Marketing.

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NUCRO TECHNICS LIMITED

2000 ELLESMERE ROAD, UNIT 16, SCARBOROUGH, ONTARIO M1H 2W4 416/438-6727

# ANALYTICAL REPORT

Mr. John Trought
Manager
Envirocon (Eastern) Limited
P.O. Box 1339
Downsview, Ontario
M3H 5W3

DATE November 26, 1979

PROJECT NO. 24769 A

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL, AND FILTERS

Procedures:

A) Cleaning of glassware in train.
Glass components of a train consisted of the following:

up to 5 impingers

up to 6 U-tubes

up to 5 connecting pieces

up to 2 Florisil filter cartridges.

These glass components were subjected to a cleaning procedure consisting of a minimum 24 hour soak in Decon 75 and tap water. Components were scrubbed by hand employing soft bristle brushes before and after the soak. Components were generously rinsed with tap water followed by a rinse with once distilled water. Components were then generously rinsed with pesticide grade acetone (2X) followed by pesticide grade hexane (2X) on the interior and connecting surface. Because of visible contamination from previous use some previously used components were subjected to more vigorous measures such as heating in the flame of a propane torch and extended soaks in methylene chloride, pesticide grade.

Glassware components that appeared to be visually contaminated at this stage were recycled thru the Decon 75 soak. Some glassware items were permanently rejected as uncleanable.

Glassware which appeared to be visually free of contamination was placed in a 250°C forced air oven for a minimum of 12 hours. Many components were held at this temperature for extended periods of time up to 5 days until sufficient components to constitute a train were available and had reached the same stage of the cleaning procedure.

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SUBJECT CLEANING & PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL, AND FILTERS

November 26, 1979

Proposal No. 24769A

After removal from the forced air oven components were allowed to cool in the open air on paper covered laboratory bench space. A final cleaning rinse to waste of the interior and connecting furnaces with pesticide grade pentane was carried out.

A subsequent rinse with pentane of all interior and connecting surfaces was carried out and collected in a 600 milliliter Kuderna Danish concentrator. Similarly, pentane rinsings from other components of the train which were cleaned by different procedures were combined in the associated Kuderna Danish Concentrator.

B) Cleaning of Glass probes.
Because of their extreme length it was not possible to heat the probes in an oven.

Probes were cleaned by generous rinsing of the interior and connecting surfaces with acetone (2X) followed by hexane (2X). When the probes were rinsed they were held at approximately a 30° angle to the horizontal and rotated about their long axis. This procedure provides an extended contact time and complete contact of the interior surface.

Visual examination of the glass probe both of the exterior and interior surfaces was necessary. Repeated rinsings were required to remove particulate visible when looking into the interior of the probe along its long axis.

Glass probes were rinsed once more with pentane prior to collecting a final pentane rinse in the associated Kuderna-Danish concentrator.

C) Cleaning of Frittered Glass Filter Support Disc
Because of the nature of the frittered glass filter support disc and its thickness it was decided not to expose it to the detergent soak. All frittered glass filter support discs employed were new.

The discs were soaked in acetone for 12 hours, generously rinsed with acetone then transferred to a soak in hexane for 12 hours followed by generous rinsing. The discs were then transferred to a 250°C forced air oven for a minimum of 12 hours. After removal from the oven the discs were allowed to cool on the paper covered laboratory bench and then generously rinsed with pentane to waste.

A final generous rinse with pentane sufficient to completely wet the frittered material was carried out while the disc was held above the Kuderna Danish concentrator and rotated.

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SUBJECT CLEANING & PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL, AND FILTERS

DATE November 26, 1979

PROJECT NO 24769

D) Nozzles
Sufficient nozzles for all trains were not available.

Because of the unsatisfactory experience with the 1st. train's externally nickel plated stainless steel nozzle an additional cleaning and partial confirmation of satisfactory internal cleanliness was introduced.

After the scrubbing and 24 hour soak in Decon 75 the nozzles were rinsed with acetone 2X and hexane 2X. A doubled over pipe cleaner soaked in hexane was introduced thru the bore of the nozzle and forced thru the nozzle while rotating the stem of the pipe cleaner.

Initial efforts employed a slow speed setting of a variable speed electric drill to impart the rotary motion necessary. However, there was a distinct fire hazard associated with the use of an electric drill and further efforts were conducted by hand.

The absence of discolouration such as grease or oil on the pipe cleaner was considered as sufficient evidence of internal "visual" cleanliness.

Nozzles were then held for a minimum of 12 hours in a 250°C forced air oven. After removal from the oven, the nozzles were allowed to cool on the paper covered bench. The interior and connecting surfaces were rinsed with pentane IX.

A final rinse of the internal and connecting surfaces with pentane IX was collected in the associated Kuderna-Danish concentrator.

E) Cleaning of Aluminum Foil and Sealing of train Components.

Commercially available heavy duty aluminum foil was cleaned up by the following procedure. Sheets of foil 24" x 16" were soaked in Decon 75 - water solution for a minimum 24 hours then rinsed with tap water followed by a rinse with IX distilled water.

The sheets were then immersed in acetone for 3 hours allowed to drain then immersed in hexane for 3 hours and allowed to drain. The foil sheets were then allowed to air dry prior to being placed in a 250°C forced air oven for a minimumof 12 hours.

..../4

SUBJECT CLEANING & PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL, AND FILTERS

DATE November 26, 1979

PROJECT NO. 24769 A

The aluminum foil was proven as a batch by rinsing both sides of 5 to 6 representative sheets with pentane which was collected in a Kuderna Danish concentrator, concentrated and assayed by Gas Liquid Chromatography (GLC).

The proven aluminum foil was employed to seal off the interior and connecting surfaces of all glassware and metal components previously cleaned. Components were not assembled into the train at Nucro-Technics but sufficient components to constitute a train were cleaned, proven together, sealed with the aluminum foil and provided to Envirocon (Eastern) Limited as a set.

F) Because of problems with the final assembly of the front and rear filter holders in the locking rings, Envirocon (Eastern) Limited returned the filter holders to the manufacturer for alteration. An abbreviated cleaning procedure was employed after return of the filter holders to Nucro-Technics Limited.

Although this operation was carried out on two occasions only the abbreviated cleaning procedure employed for the final release is reported.
The filter holders received a hand scrub with soft bristle brushes and were soaked in Decon 75 - water solution for four hours. The holders were generously rinsed with tap water followed by a rinse with IX distilled water. The holders were then rinsed with acetone 2X, followed by hexane 2X, and baked out at 250° in a forced air oven for 9 hours. After removal from the oven the holders were allowed to cool and rinsed with pentane 2X.

A final rinse with pentane IX was collected in a Kuderna-Danish concentrator for subsequent GLC assay.

In review, the filter holders released with the trains were subsequently modified, cleaned by an abbreviated procedure as described and proven as a batch prior to final release.

G) Cleaning and proving of glass fiber filters.

Glass fibre filters of a nominal 5 inch diameter were provided by Envirocon (Eastern) Limited.

The filters were cleaned by soxhlet extraction employing hexane. A medium sized soxhlet apparatus was operated for 12 hours per day for three days at a cycle rate of 2 cycles per hour.

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SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL, AND FILTERS

DATE November 26, 1979

PROJECT NO. 24769 A

After soxhlet extraction the filters were placed in a 120°C oven overnight prior to weighing. After weighing individual filters were packaged in precleaned aluminum foil and assigned to a train.

Two filters cleaned by the above procedure and held in the 120°C oven were extracted in a Soxhlet apparatus with pentane for 8 hours operating at a cycle rate of 3 cycles per hour.

The pentane extract was quantitatively transferred to a Kuderna-Danish concentrator for concentration and subsequent GLC assay.

H) Cleaning, Preparation and Proving of Florisil Cartridges.

The glass cartridges were cleaned as described in section A but were not proven with the train.

After cleaning the unpacked cartridges were held at 250°C in the forced air oven. Each cartridge was engraved with a number from 2 to 16 on the body of the cartridge closest to the inlet end.

Glass wool was cleaned by extraction with pentane in a beaker. After pouring off the excess pentane, the beaker containing the glass wool was placed at the rear of a fume hood and allowed to dry. When dry, the beaker and glass wool was transferred to a 120°C oven for storage.

A portion of the glass wool cleaned and stored as above was extracted by shaking in pentane for 5 minutes. The pentane extract was collected in a Kuderna-Danish concentrator for concentration and subsequent GLC assay.

Florisil 30/60 mesh was cleaned in a tube furnace as a batch. The process employed heats the material to 650°C over a 12 hour period after which it is held at 650°C for a minimum of 12 hours and then allowed to cool for an additional 12 hours. During the heating process florisil filtered air under pressure purges the glass column. After removal from the tube furnace the florisil is held in a 120°C oven until use.

Individual cartridges were filled with florisil prepared as above. A layer of glass wool prepared as above is tamped in place with a glass rod over the florisil.

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SUBJECT CLEANING & PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL, AND FILTERS

DATE November 26, 1979

PROJECT NO. 24769 A

Each cartridge was eluted with 2 X 100 milliliter portions of 10% methylene chloride in pentane and allowed to drain. This was followed by 2 X 100 milliliter portions of pentane and allowed to drain. A final eluate of 100 milliliter pentane was collected in individual Kuderna-Danish concentrators for each cartridge. The eluate was concentrated and assayed by GLC.

In review the components of the florisil cartridges were cleaned up individually, then assembled or packaged with a final cleaning procedure of the assembly followed by an individual proving of each florisil cartridge.

After elution with pentane the cartridges were blown down with florisil filtered air, then reactivated by a minimum 48 hour holding period in a 120°C oven. After removal from the oven the ends of the florisil cartridge were sealed with a double layer of proven aluminum foil.

1) Concentration of Pentane for GLC Assay.

Pentane from a proving rinse or extraction was collected in, or transferred to a Kuderna-Danish concentrator. A keeper of 0.5 milliliter iso-octane and two or three precleaned boiling chips were added.

Samples were concentrated to approximately 5 milliliters employing the main body of the Kuderna-Danish concentrator and a three ball macro Snyder column on a water bath at 60°C.

The main body of the Kuderna-Danish was removed and a macro Snyder column substituted for the macro Schneider. The concentration was continued on a heating apparatus to 0.5 milliliters or less, then made up to 0.5 milliliters.

The iso-octane concentrate was transferred to precleaned glass micro vials with aluminum foil lined screw caps for holding until GLC assay.

Results of GLC Assay

Table 1 - Trains for In Lab Proving Run

| Part | Concentration PCB ng/ml | Total PCB in training |
|---|---|---|
| Train 1 | excessive | excessive |
| Train 2 | 69.03 | $\leq$34.5 |

SUBJECT CLEANING & PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL, AND FILTERS

November 26, 1979

24769 A

Results of GLC Assay cont'd:

Table 2 - Trains for Preburn Stack Sampling.

| Part | Concentration PCB ng/ml | Total PCB in train ng |
|---|---|---|
| Train 3 | 9.73 | ≤ 4.9 |
| Train 4 | 48.97 | ≤ 24.5 |
| Train 5 | 32.64 | ≤ 16.3 |
| Train 6 | 3.48 | ≤ 1.7 |
| Train 7 | 8.68 | ≤ 4.3 |
| Train 8 | 10.39 | ≤ 5.2 |
| Supplemental #2 Filter Holders | 36.4 | ≤ 18.2 |

Table 3 - Florisil Cartridges

| Part | Concentration PCB ng/ml | Total PCB in Part ng |
|---|---|---|
| Florisil cartridge 1 | 9.36 | ≤ 4.7 |
| Florisil cartridge 2 | 38.87 | ≤ 19.4 |
| Florisil cartridge 3 | recalled | recalled |
| Florisil cartridge 4 | recalled | recalled |
| Florisil cartridge 5 | recalled | recalled |
| Florisil cartridge 6 | recalled | recalled |
| Florisil cartridge 7 | recalled | recalled |
| Florisil cartridge 8 | recalled | recalled |
| Florisil cartridge 9 | 42.25 | ≤ 21.1 |
| Florisil cartridge 10 | 6.25 | ≤ 3.1 |
| Florisil cartridge 11 | 68.46 | ≤ 32.2 |
| Florisil cartridge 12 | 3.81 | ≤ 1.9 |
| Florisil cartridge 13 | 258.2 | ≤ 129. |
| Florisil cartridge 14 | 16.73 | ≤ 8.4 |
| Florisil cartridge 15 | 46.98 | ≤ 23.5 |

SUBJECT CLEANING & PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL, AND FILTERS

DATE November 26, 1979

PROJECT NO 24769 A

Results of GLC Assay cont'd:

Table 4 - Ancillary Supplies

| Part | Concentration PCB's ng/ml | Total PCB ng |
|---|---|---|
| Aluminum Foil | 20.98 | 21.0 (6 pieces) |
| Glass wool | | None detected |
| Aluminum Foil | 84.32 | 84.32(6 pieces) |
| Glass Fibre Filters | 0.79 | 0.4 (2 pieces) |

The results for GLC assay for trains 1 to 8 and florisil cartridges 1 to 15 are presented in tabular format, tables 1-4.

Fred S. Seymour, B.Sc.
Director Technical Marketing

jc

NUCRO TECHNICS LIMITED

ANALYTICAL REPORT

2000 ELLESMERE ROAD, UNIT 16, SCARBOROUGH, ONTARIO M1H 2W4 416/438-6727

John Trought, P. Eng.
General Manager
Envirocon (Eastern) Limited
P.O. Box 1339
Downsview, Ontario
M3H 5W3

DATE January 18th, 1980

PROJECT NO 24769 D1

SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

Procedures:

A) Disassembly of Trains

Stack sampling trains were transported intact as assembled for the stack testing by Envirocon (Eastern) Limited (EEL) personnel to Nucro-Technics Limited. The intake and outlet openings were sealed after the completion of the on stack portion of the test by EEL personnel. Precleaned and proven aluminum foil provided by Nucro-Technics Limited (NTL) was employed for the sealing of the trains.

Trains were partially disassembled prior to extraction. As each section was removed from the train the inlet and outlet openings were sealed with aluminum foil. Seals were secured with light metal wire twisted tight. Additionally each major component of the train was labelled with a tag as to component number and train. All components with the exception of glass probes were stored together until extraction.

B) Sample Recovery and Extraction

(i) Glass Sampling probe, nozzle, cyclone bypass and front section of filter holder.

In order to maximize the recovery of particulate from the probe but prevent contamination from nylon, plastic or natural hair embedded in plastic or epoxy resins the use of all metallic shotgun cleaning brushes was proposed.

Shotgun brushes with bristles of brass in 0.410 inch bore size were found to be suitable. Cleaning and proving procedures are covered in the material section of this report.

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SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

The shotgun brushes were employed with an eight foot handle to clean the internal bore of the seven foot glass sampling probe. Two passes of the brush were found to be necessary. After the brushing procedure the brush was rinsed with pesticide grade pentane. The interior surfaces of the probe was rinsed with successive washes of pentane while the probe was slowly rotated.

The pentane washes and recovered particulate were collected in a previously cleaned and proven glass container.

The interior surfaces of the nozzle, cyclone bypass and front filter holder were generously rinsed (four rinses) with pentane. The pentane rinses and recovered particulate were combined with the probe rinses.

A fifth and final rinse of all interior surfaces of the probe, nozzle, cyclone bypass and front filter holder was conducted and combined in a seperate, previously cleaned and proven container.

The total combined first four rinses were filtered through a previously cleaned pretared proven fiberglass filter. The pentane filtrate was collected in a seperate previously cleaned and proven glass container.

The filter and residue was dried in a precleaned glass and metal dessicator for twenty four hours and weighed. The filter was then extracted in a soxhlet apparatus for a minimum of 8 hours operating at a cycle rate of 3 per hour.

The initial pentane charge (∿200 ml) and two (∿30 ml) rinses of the receiver were combined with filtrate previously described.

(ii) Glass Fiber Filters

After removal from the filter holder the filters were supported by the frittered glass filter support disc. The filter with supporting disc was placed in a previously cleaned glass and metal dessicator over fresh silica gel for twenty four hours. After conditioning the filter and residue was weighed. See Table 1 - Filter Residues.

The filter and residue was then extracted in a Soxhlet apparatus employing an initial charged ∿200 ml pentane. The filter and residue was extracted for a minimum of 8 hours at a cycle rate of three per hour. The initial pentane charge and two (∿30 ml) pentane rinses of the receiver were combined in a previously cleaned and proven glass container.

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SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

Table 1 - Filter Residue

| Sample Designation | Final Weight | Initial Weight | Residue Weight |
|---|---|---|---|
| Train A, Kiln | 1.9501g | 1.8059g | 0.1442g |
| Train B, Kiln | 0.9885 | 0.0792g | 0.0792 |
| Clinker Cooler | 0.9059 | 0.8999 | 0.0060 |
| Blank | 0.9079 | 0.9046 | 0.0033 |

...../4

SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

(iii) Frittered Glass Filter Support and Rear Filter Holder

The frit was extracted by soaking in pentane in a foil covered beaker overnight. Two additional rinses with pentane sufficient to soak the frit were combined with the first extract and the total extract was transferred to a previously cleaned and proven glass container.

The interior surfaces of the rear half of the filter holder were rinsed three times and the rinses combined with the frit extract.

A final rinse of the frit and rear filter holder was collected in a separate precleaned and proven glass container.

(iv) Impinger Connecting Glassware

Impinger connecting glassware with visible water droplets was rinsed with two one ml portions of pesticide grade acetone per piece. Four rinses with pentane of the interior surfaces of each piece were conducted and combined with the acetone in a precleaned proven glass container. A final rinse with pentane of each piece was collected in a separate precleaned and proven glass container.

(v) Impinger Contents

Water for impinger pre-loading was supplied by the Ministry of Environment. Pre-sampling water volume in impingers 1 and 2 was determined by Envirocon (Eastern) Limited.

Each individual impinger for all five trains was preweighed and labelled by Envirocon (Eastern) Limited. After receipt the total weight of each individual impinger was determined by Nucro-Technics Limited. See Table 2 - Impinger Contents.

The total water content of all impingers was equally distributed between all of the impingers of a train. A nominal 100 ml of pentane was added to each impinger and the impinger and contents was extracted by vigorous shaking for a minimum of five minutes. After shaking the phases were allowed to separate and the pentane (upper phase) drawn off by aspiration employing a long tipped 25 or 50 ml pipette and a rubber bulb. The extraction was repeated four times with each extract being successively filtered through anhydrous sodium sulfate retained in a proven glass funnel with proven glass wool. The impinger pentane extracts for a train were combined with the impinger connecting glassware extract.

In some cases the total volume of pentane extract of the impingers exceeded one litre and an additional proven glass container was necessary.

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SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

(vi) Extraction of Florisil Cartridges

The entire contents of the cartridge including glass wool was expelled into a precleaned and batch proven cellulose thimble. The florisil and glass wool were extracted in a Soxhlet extraction apparatus for a minimum of eight hours at a cycle rate of three per hour. The initial pentane change of ∿200 ml and two ∿30 ml rinses of the receiver were combined in a cleaned and proven glass container.

C) Materials

Solvents - Pesticide Grade, Glass Distilled; Pentane, Acetone, Methylene Chloride, Hexane, Iso-octane from Caledon Laboratories.
HPLC Grade Methanol from BDH Chemicals

Glass Wool - Previously cleaned and proven. See #24769 A

Anhydrous Sodium Sulfate:

Anhydrous sodium sulfate ACS grade (Canlab) was cleaned in a vertical tube furnace as a batch. The process employed heats the material to 650°C over a 12 hour period after which it is held at 650° for a minimum of 12 hours and then allowed to cool for an additional 12 hours. During the heating process florisil filtered air under pressure purges the quartz column containing the material. After removal from the tube furnace the florisil is held in a 120°C oven until use.

Proving of sodium sulfate was undertaken by filtering 200 ml of pentane through 15g of sodium sulfate contained in a precleaned glass funnel. The pentane eluate-filtrate was concentrated for GLC assay as previously reported. See report #24769 A.

D) Cleaning and Proving of Glassware and Final Containers for Extracts.

Glassware and glass containers for the pentane extracts were cleaned by an extensive cleaning process. A minimum 12 hour soak in Decon 75 - water was followed by generous rinsing with tap water, followed by distilled water. Solvent rinsings in sequence were methylene chloride IX, acetone 2X, and hexane 2X. After air drying glassware was baked in a 250°C forced air oven for a minimum of twelve hours. After cooling a rinse with pentane to waste was conducted.

A final rinse with pentane ∿15 ml per piece or more was collected and concentrated for GLC assay.

...../6

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXP S ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: .....
Source: ..... Region: .....
Program: .....
Date sampled: ..... by: .....
Date analysed: ..... Date reported: .....

Report to: .....
Address: .....

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | √CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| Eq 3685 | | B Kiln 1st, 2nd, 3rd Pentane Rinses ~ Rear Half of filter Holder. | | |
| Eq 3686 | | ✓ ✓ ✓ ✓ ✓ ✓ ✓ Probe, Nozzle, rear half of filter. | | |
| Eq 3687 | | ✓ ✓ FLORISIL CARTRIDGE #14 SOXHLET EXTRACTION | | |
| Eq 3688 | | ✓ ✓ Probe Rinse and Filter Extraction | | |
| Eq 3689 | | 6 Pieces impingers connecting glassware | | |
| Eq 3690 | | B Kiln 4 pentane rinse of probe, Nozzle and front half of filter. | | |
| Eq 3691 | | ✓ ✓ 4th Rinsing ~ 6 pieces impingers connecting glassware. | | |
| Eq 3692 | | ✓ ✓ Rear half of filter holder. | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | RB [illegible] Blk/1980 TOTAL WT. | | | | | | | | | |
| 54 | | | | | | | | | | | |
| 55 | | | | | | | | | | | |
| 56 | | | | | | | | | | | |
| 57 | | 1980 | | | | | | | | | |
| 58 | | X | | | | | | | | | |
| 59 | | | | | | | | | | | |
| 60 | | | | | | | | | | | |
| 61 | | | | | | | | | | | |
| 62 | | | | | | | | | | | |

MOE 0951

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESC CES ROAD
HIGH .Y 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: .....
Source: ..... Region: .....
Program: .....
Date sampled: ..... by: .....
Date analysed: ..... Date reported .....

Report to: .....
Address: .....

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

"✓" = YES
"—" = NO

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT | DE | TAPE |
|---|---|---|---|---|---|---|
| [illegible] 3693 | | Kiln A Train #8 Probe, Nozzle, front Half of filter holder (4th Rinse) | | | ✓ | ✓ |
| [illegible] 3694 | | ✓ ✓ ✓ Rear Half of filter holder 1st 3 rinses | | | ✓ | ✓ |
| [illegible] 3695 | | ✓ ✓ ✓ 1st, 2nd, 3rd, Pentane Rinses from Probe, Nozzle, front half of filter | | | ✓ | — |
| [illegible] 3696 | | ✓ ✓ ✓ impinger connecting glassware 5 pieces 4th Rinsing | | | ✓ | — |
| [illegible] 3697 | | ✓ ✓ ✓ 4th Pentane Rinse of rear half of filter holder. | | | ✓ | — |
| [illegible] 3698 | | A Kiln ✓ ✓ Probe Rinse and filter Extraction. | | | — | ✓ |
| [illegible] 3699 | | ✓ ✓ ✓ Cartridge # 15 | | | — | ✓ |
| | | | | | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | PCB's Total | as Aroclor 1242/1260 mg/l | | | | | | | | |
| [illegible] 3693 | | | | | | | | | | | |
| [illegible] 3694 | | | | | | | | | | | |
| [illegible] 3695 | | | | | | | | | | | |
| [illegible] 3696 | | | | | | | | | | | |
| [illegible] 3697 | | | | | | | | | | | |
| [illegible] 3698 | | | | | | | | | | | |
| [illegible] 3699 | | 0 | | | | | | | | | |
| [illegible] 3700 | | 3.50 | | | | | | | | | |
| | | | | | | | | | | | |

MOE 0951

Sample no. PCB-3674

Location ST LAWRENCE CEMENT PLANT

BLANK TRAIN FLORISIL CARTRIDGES #17

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | 120.4 | 45.6 | 33.4 | 47.5 | 9.9 | 29.9 | |
| 4 | 120.8 | | | | | | | |
| 26 | 0.4 | | | | | | | |
| 22' | 11.0 | | | | | | | |
| 25 | 18.0 | | | | | | | |
| 23 | 4.3 | | | | | | | |
| 246 | 0.6 | | | | | | | |
| 225 | 11.4 | | | | | | | |
| 44' | 12.3 | | | | | | | |
| 2246 | 1.8 | | | | | | | |
| 245 | 21.6 | | | | | | | |
| 2256' | 10.4 | | | | | | | |
| 2346 | 0.2 | | | | | | | |
| 2255' | 9.3 | | | | | | | |
| 2235' | 9.6 | | | | | | | |
| 22446 | 5.4 | | | | | | | |
| 2233' | 5.2 | | | | | | | |
| 2345 | 10.8 | | | | | | | |
| 22455' | 4.5 | | | | | | | |
| 23456 | | | | | | | | |
| 22336' | 2.6 | | | | | | | |
| 3344' | 0.2 | | | | | | | |
| 2244'5' | 1.2 | | | | | | | |
| 22345 | 0.1 | | | | | | | |
| 223455'6 | | | | | | | | |
| 2233'44' | 1.4 | | | | | | | |
| 2334'45 | 24.6 | | | | | | | |
| Total | 257.7 | | | | | | | |

Sample no. P-3177

Location ST LAWRENCE CEMENT PLANT

BLANK FOR FIBREGLASS FILTERS.

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | 46.9 | 56.6 | 16.7 | 29.5 | 6.5 | 2.4 | |
| 4 | 46.9 | | | | | | | |
| 26 | 0.2 | | | | | | | |
| 22' | 10.3 | | | | | | | |
| 25 | 10.4 | | | | | | | |
| 23 | 9.8 | | | | | | | |
| 246 | 1.4 | | | | | | | |
| 225 | 5.3 | | | | | | | |
| 44' | 25.9 | | | | | | | |
| 2246 | 1.6 | | | | | | | |
| 245 | 10.0 | | | | | | | |
| 2256' | 5.5 | | | | | | | |
| 346 | | | | | | | | |
| 2255' | 5.6 | | | | | | | |
| 2235' | 5.2 | | | | | | | |
| 2246 | 3.1 | | | | | | | |
| 2233' | 2.9 | | | | | | | |
| 2345 | 7.5 | | | | | | | |
| 2245'5' | 3.2 | | | | | | | |
| 23456 | 0.2 | | | | | | | |
| 2233'66' | 3.1 | | | | | | | |
| 33'44' | 1.54 | | | | | | | |
| 2244'55' | 0.8 | | | | | | | |
| 2344'5 | 0.4 | | | | | | | |
| 23455'6 | | | | | | | | |
| 2233'44' | 0.8 | | | | | | | |
| 2334'45 | 36.3 | | | | | | | |
| Total | 197.6 | | | | | | | |

Sample no. P.S.-3631

Location ST LAWRENCE CEMENT PLANT
CLINKER COOLER. TRAIN 7
CARTRIDGE #9.

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | 41.9 | 141.4 | 50.7 | 54.9 | 3.9 | 21.7 | |
| | 41.9 | | | | | | | |
| 26 | 59.9 | | | | | | | |
| 22' | 50.8 | | | | | | | |
| 25 | 14.7 | | | | | | | |
| 23 | 5.7 | | | | | | | |
| 246 | 21.5 | | | | | | | |
| 225 | 16.0 | | | | | | | |
| 44' | 4.7 | | | | | | | |
| 2246 | 23.5 | | | | | | | |
| 245 | 11.9 | | | | | | | |
| 2256' | 0.1 | | | | | | | |
| 346 | 14.5 | | | | | | | |
| 2255' | 10.2 | | | | | | | |
| 2235' | | | | | | | | |
| 2246 | 3.5 | | | | | | | |
| 2233' | 9.4 | | | | | | | |
| 2345 | 3.7 | | | | | | | |
| 2245' | 0.1 | | | | | | | |
| 2356 | 2.4 | | | | | | | |
| 22336' | 0.6 | | | | | | | |
| 334' | 0.5 | | | | | | | |
| 2244'5' | 1.7 | | | | | | | |
| 22344 | | | | | | | | |
| 223456 | | | | | | | | |
| 22334' | 0.7 | | | | | | | |
| 23344 | 20.5 | | | | | | | |
| Total | 318.5 | | | | | | | |

Sample no. P4-3654

Location ST LAWRENCE CEMENT PLANT
CLINKER COOLER TRAIN 7
PENTANE RINSE FROM FILTER

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 8.1 | 76.4 | 68.1 | 16.4 | 32.4 | 5.3 | 6.1.6 | |
| 4 | 65.3 | | | | | | | |
| 26 | | | | | | | | |
| 22' | 19.2 | | | | | | | |
| 25 | 12.7 | | | | | | | |
| 23 | 11.2 | | | | | | | |
| 246 | 2.9 | | | | | | | |
| 225 | 4.9 | | | | | | | |
| 44' | 25.0 | | | | | | | |
| 2246 | 1.6 | | | | | | | |
| 245 | 8.6 | | | | | | | |
| 2256' | 5.0 | | | | | | | |
| 2,46 | | | | | | | | |
| 2255' | 6.9 | | | | | | | |
| 2235' | 5.4 | | | | | | | |
| 2246 | 0.2 | | | | | | | |
| 2233' | 2.4 | | | | | | | |
| 2345 | 6.1 | | | | | | | |
| 2255' | 7.1 | | | | | | | |
| 23456 | | | | | | | | |
| 223366' | 6.3 | | | | | | | |
| 3344' | 1.0 | | | | | | | |
| 2244'55' | 2.4 | | | | | | | |
| 22344'5 | 0.1 | | | | | | | |
| 223455'6 | | | | | | | | |
| 2233'44' | | | | | | | | |
| 2334'45 | 32.6 | | | | | | | |
| Total | 239.1 | | | | | | | |

Sample no. P-g-3657

Location St Lawrence cement

TB Kiln Florisil Cartridge

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | 45.6 | 257.3 | 76.7 | 77.6 | 34.0 | 26.4 | 5.3 |
| 4 | 45.6 | | | | | | | |
| 26 | 168.2 | | | | | | | |
| 22' | 940.6 | | | | | | | |
| 25 | 441.4 | | | | | | | |
| 23 | 266.8 | | | | | | | |
| 246 | 29.0 | | | | | | | |
| 225 | 464.0 | | | | | | | |
| 44' | 755.8 | | | | | | | |
| 22'46 | | | | | | | | |
| 245 | 274.0 | | | | | | | |
| 2256' | 185.5 | | | | | | | |
| 2' 46 | | | | | | | | |
| 22'55' | 254.6 | | | | | | | |
| 22'35' | 145.1 | | | | | | | |
| 22'446 | 64.7 | | | | | | | |
| '22'33' | 28.0 | | | | | | | |
| 23'45 | 143.5 | | | | | | | |
| 22455' | 116.5 | | | | | | | |
| 23456 | 158.6 | | | | | | | |
| 22'33'66' | 27.5 | | | | | | | |
| 33'44' | 9.4 | | | | | | | |
| 22'44'55' | 78.6 | | | | | | | |
| 22'344'5 | 34.5 | | | | | | | |
| 22'3455'6 | 5.3 | | | | | | | |
| 22'33'44' | 44.1 | | | | | | | |
| 233'44'5 | 78.9. | | | | | | | |
| Total | 4771 | | | | | | | |

Sample no. P29-3628

Location ST Lawrence cement
'B' KILN PRIOR / FILTER HOUSE

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 31.7 | | | | | | | |
| 4 | 93.6 | | | | | | | |
| 6 | 1.7 | | | | | | | |
| 22' | 32.1 | | | | | | | |
| 25 | 15.4 | | | | | | | |
| 23 | 12.4 | | | | | | | |
| 246 | 5.1 | | | | | | | |
| 225 | 12.4 | | | | | | | |
| 44' | 81.4 | | | | | | | |
| 2246 | 1.8 | | | | | | | |
| 245 | 8.1 | | | | | | | |
| 2256' | 3.6 | | | | | | | |
| 2346 | | | | | | | | |
| 255' | 12.0 | | | | | | | |
| 2235' | 8.6 | | | | | | | |
| 2246 | 2.4 | | | | | | | |
| 2235' | 2.0 | | | | | | | |
| 2345 | 12.9 | | | | | | | |
| 2245' | 11.8 | | | | | | | |
| 23456 | 0.3 | | | | | | | |
| 2235'6' | 17.2 | | | | | | | |
| 3344' | 1.9 | | | | | | | |
| 2244'5' | 6.6 | | | | | | | |
| 2234'45 | 3.4 | | | | | | | |
| 2234'5'6 | 0.1 | | | | | | | |
| 2233'44' | 1.4 | | | | | | | |
| 2333'445 | 20.3 | | | | | | | |
| Total | 400.1 | | | | | | | |

Sample no. 29-3698

Location St Lawrence estuary
'A' Island. Probe Plume Study.

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 36.5 | | | | | | | |
| 4 | 67.2 | | | | | | | |
| 2[illegible] | | | | | | | | |
| 22' | 21.5 | | | | | | | |
| 25 | 11.6 | | | | | | | |
| 23 | 6.6 | | | | | | | |
| 246 | 1.5 | | | | | | | |
| 225 | 10.1 | | | | | | | |
| 44' | 107.9 | | | | | | | |
| 2246 | 5.5 | | | | | | | |
| 245 | 12.6 | | | | | | | |
| 2256' | 9.5 | | | | | | | |
| 2346 | | | | | | | | |
| 2[illegible]35' | 16.5 | | | | | | | |
| 22'35' | 11.1 | | | | | | | |
| 22'446 | 3.9 | | | | | | | |
| 22'33' | 4.3 | | | | | | | |
| 23'45 | 18.0 | | | | | | | |
| 22'455' | 15.9 | | | | | | | |
| 23456 | 0.2 | | | | | | | |
| 22'33'66' | 23.7 | | | | | | | |
| 33'44' | 1.2 | | | | | | | |
| 22'44'55' | 19.5 | | | | | | | |
| 22'344'5 | 3.0 | | | | | | | |
| 22'3455'6 | 0.2 | | | | | | | |
| 22'33'44' | 11.3 | | | | | | | |
| 233'44'5 | 33.7 | | | | | | | |
| Total | 453.0 | | | | | | | |

Sample no. 229-3699

Location ST LAWRENCE RIVER
50' MILE [illegible]

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 2553.1 | | | | | | | |
| 4 | 627.9 | | | | | | | |
| [illegible] | 45.1 | | | | | | | |
| 22' | 658.1 | | | | | | | |
| 25 | 7.0 | | | | | | | |
| 23 | 79.9 | | | | | | | |
| 246 | 6.8 | | | | | | | |
| 225 | 36.3 | | | | | | | |
| 44' | 362.7 | | | | | | | |
| 2246 | 23.0 | | | | | | | |
| 245 | 32.8 | | | | | | | |
| 2256' | 24.9 | | | | | | | |
| 2346 | | | | | | | | |
| 2'55' | 52.0 | | | | | | | |
| 22'35' | 40.2 | | | | | | | |
| 22'446 | 15.0 | | | | | | | |
| 22'33' | 12.3 | | | | | | | |
| 234'5 | 60.6 | | | | | | | |
| 22'455' | 56.8 | | | | | | | |
| 23456 | 0.3 | | | | | | | |
| 22'33'66' | 95.2 | | | | | | | |
| 33'44' | 4.9 | | | | | | | |
| 22'44'55' | 53.8 | | | | | | | |
| 22'344'5 | 20.4 | | | | | | | |
| 22'3455'6 | 3.0 | | | | | | | |
| 22'33'44' | 39.7 | | | | | | | |
| 233'44'5 | 74.1 | | | | | | | |
| Total | 4995.2 | | | | | | | |

SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

Cellulose thimbles for Soxhlet extraction were cleaned by Soxhlet extraction. A large Soxhlet apparatus was operated for up to 15 hours per day at a cycle rate of 1 cycle per 3 hours. Initial solvent charge was 3.5 litres hexane. Operation of the apparatus for in excess of two weeks was necessary before a successful proving assay was obtained. Chromatograms of proving run failures have been included.

Approximately 20 thimbles were cleaned up in a batch. Approval for use of the batch was given after two thimbles from the top of the load gave an acceptable proving chromatogram on GLC assay. The two precleaned thimbles were extracted in a Soxhlet apparatus with pentane for 8 hours at a cycle rate of three per hour. The extracts were combined and concentrated for GLC assay. Additional proving runs for thimbles cleaned up in the same batch are included in report #24769 E.

...../7

SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

Table 2 - Impinger Contents

| Impinger # & Train | Final Weight | Initial Weight | Net $H_2O$ Gain |
|---|---|---|---|
| Train A, Kiln | | | |
| 3 - 1 | 871.9g | 564.5g | 307.4 |
| 3 - 2 | 932.6 | 561.0 | 371.6 |
| 3 - 3 | 955.8 | 481.6 | 474.2 |
| 3 - 4 | 493.5 | 475.6 | 17.9 |
| 3 - 5 | 478.4 | 472.7 | 5.7 |
| | | Total | 1176.8g |
| Train B, Kiln | | | |
| 5 - 1 | 781.6g | 569.0g | 212.6g |
| 5 - 2 | 844.2 | 563.8 | 280.4 |
| 5 - 3 | 853.3 | 481.2 | 372.1 |
| 5 - 4 | 489.6 | 478.9 | 10.7 |
| 5 - 5 | 474.4 | 472.3 | 2.1 |
| | | Total | 877.9g |
| Clinker Cooler | | | |
| 7 - 1 | 566.8g | 539.1g | 27.7g |
| 7 - 2 | 601.9 | 573.5 | 28.4 |
| 7 - 3 | 488.1 | 475.7 | 12.4 |
| | | Total | 68.5g |
| Blank | | | |
| 6 - 1 | 581.0g | 581.0g | 0g |
| 6 - 2 | 569.1 | 569.6 | - 0.5 |
| 6 - 3 | 467.1 | 459.5 | 7.6 |
| 6 - 4 | 470.8 | 458.6 | 12.2 |
| 6 - 5 | 485.7 | 469.4 | 16.3 |
| | | Total | 35.6g |

..../8

SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

Results for GLC assay for materials and glassware employed in sample extraction are presented in Table 3.

Table 3 - Total PCB Content of Materials and Glassware Employed in Sample Extraction.

| Part | Concentration Total PCB ng/ml | Total PCB in Part ng |
|---|---|---|
| Shotgun Brushes | None Detected | None Detected for 2 |
| Sodium Sulfate | None Detected | None Detected for 15 grams |
| Thimbles (3rd Proving) | $\leq$ 64.624 | $\leq$ 32.3 for two |
| 25 ml Pipettes | None Detected | None Detected |
| Foil | $\leq$ 13.76 | $\leq$ 6.7 for 5 pieces |
| Glass Funnels | $\leq$ 0.56 | $\leq$ 0.3 for 12 pieces |
| Beakers & Pipettes | $\leq$ 2.38 | $\leq$ 1.2 for 6 pieces |
| 32oz Bottles Batch 1 | $\leq$ 0.320 | $\leq$ 0.16 for 12 pieces |
| 32 oz. Bottles Batch 2 | None Detected | None Detected for 12 pieces |

...../9

SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

Extraction of Process Samples.

Kiln #1 Clinker Daily Blend.

This sample had been composited and ground by St. Lawrence Cement personnel. The sample was mixed by rotation and inversion in its original container. A 16.47g portion of the sample was extracted in a Soxhlet apparatus for a minimum of 8 hours at a cycle rate of 3 per hour.

Thimbles were previously cleaned and batch proven. The initial pentane charge of ∿200 ml and two ∿ 30 ml rinses of the receiver were collected in a previously clean and proven glass container.

Gravel Bed

The sample was mixed by rotation and inversion in its original container. A 15.03g portion of the sample was Soxhlet extracted as previously described.

Powdered Coal

This sample had been composited and ground by St. Lawrence Cement personnel. The sample was mixed by rotation and inversion in its original container. A 18.05g portion of the sample was Soxhlet extracted as previously described.

Composited Dust Sample (Precipator Dust Return)

Six samples were submitted. A composite sample consisting of equal portions by weight was made up. After mixing and rotation a 17.20g portion of the composite was Soxhlet extracted as previously described.

Composited Pelletizer Silo

Six samples were submitted. A composite sample consisting of equal portions by weight was made up. After mixing and rotation a 17.50g portion of the composite was Soxhlet extracted as previously described.

...../10

SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

Feed Slurry Composite

The feed slurry composite was made by EEL personnel. One hundred ml of sample was filtered thru a precleaned and batch proven fiberglass filter. A small quantity of methanol was poured over the residue to assist in the removal of water. The filtrate was collected ($H_2O$ and methanol) and extracted 4 X 's by liquid-liquid extraction versus pentane. The filter and residue was weighed wet and then dried in a dessicator for 48 hours and reweighed.

| | |
|---|---|
| Wet Weight Filter & Residue | 95.45g |
| Dry Weight Filter & Residue | 85.49 |
| Dry Weight of Filter | 0.887g |

| | |
|---|---|
| Wet Weight of Residue | 94.56g |
| Dry Weight of Residue | 84.60g |
| Volume of Filtrate Water + Methanol | 22 ml |

The entire residue and filter was Soxhlet extracted for a minimum of eight hours at a cycle rate of three per hour. No thimble was employed, the drain of the extraction apparatus was plugged with a pledget of previously proven glass wool so that the entire sample could be extracted. The extracts from liquid-liquid extraction of the filtrate and Soxhlet extraction of the filter and residue were combined in a clean and proven glass container.

N.B. All pentane extracts were collected in proven glass containers employing a proven foil liner. All extracts were hand delivered to the Ministry of Environment Laboratories on Resources Road.

Difficulties were experienced with evaporation of pentane from the containers. Containers were provided by the Ministry of Environment. A number of sample extracts suffered total evaporation of the pentane.

Samples which had total evaporation of pentane solvent are listed.

...../11

SUBJECT EXTRACTION OF FIVE STACK SAMPLING TRAINS AND PROCESS SAMPLES FROM FIRST PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 18, 1980

PROJECT NO. 24769 D1

Blank Train - Final Rinse of probe, nozzle, cyclone bypass and front filter holder.

Blank Train - Final Rinse of rear filter holder.

Train A, Kiln - Combined pentane rinses filtered of probe, nozzle, cyclone bypass and front filter holder.

Clinker Cooler - Combined pentane rinse filtered of probe, nozzle, cyclone bypass and front filter holder.

Train B, Kiln - Combined pentane rinses filtered of probe, nozzle, cyclone bypass and front filter holder.

Clinker Cooler - Final rinse of rear filter holder.

Clinker Cooler - Combined pentane rinses of rear filter holder.

Blank Train - Combined pentane rinses of rear filter holder.

Clinker Cooler - Final rinse of impinger connecting glassware.

Blank Train - Final rinse of impinger connecting glassware.

Train A, Kiln - Final rinse of impinger connecting glassware.

Train A, Kiln - Final rinse of rear half of filter holder

Train B, Kiln - Final rinse of impinger connecting glassware.

Train B, Kiln - Combined pentane rinses of rear filter holder.

Fred S. Seymour, B.Sc.
Director of Technical Marketing.

NUCRO TECHNICS LIMITED

ANALYTICAL REPORT

2000 ELLESMERE ROAD, UNIT 16, SCARBOROUGH, ONTARIO M1H 2W4 416/438-6727

DATE January 21, 1980

PROJECT NO. 24769 C

John Trought, P. Eng.
Manager
Envirocon (Eastern) Limited
P.O. Box 1339,
Downsview, Ontario
M3H 5W3

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL AND FILTERS

Procedures :

A) Cleaning of Glassware in a Train.

Glass components of a train consisted of the following:

up to 5 impingers
up to 6 U tubes
up to 5 connecting pieces
up to 2 Florisil filter cartridges

These glass components were subjected to a cleaning procedure consisting of a minimum 12 hour soak in Decon 75 and tap water. Components were scrubbed by hand employing soft bristle brushes before and after the soak. Components were generously rinsed with tap water followed by a rinse with once distilled water. Components were then generously rinsed with acetone (2X), followed by methylene chloride (2X), followed by hexane (2X) on the interior and connecting surfaces. Some glassware items were subjected to more vigorous measures such as extended soaks and scrubbing in methylene chloride.

Glassware components that appeared to be visually contaminated at this stage were recycled through the Decon 75 and tap water soak. As in report 24769 A, some items were permanently rejected as uncleanable.

Glassware which appeared to be visually free of contamination was placed in a 250°C forced air oven for a minimum of 12 hours. Many components were held at this temperature for extended periods of time up to four days until sufficient components to constitute a train were available and had reached the same stage of the cleaning procedure.

After removal from the forced air ovens components were allowed to cool in the open air on paper covered laboratory bench space. A final cleaning rinse to waste of the interior and connecting surfaces with pesticide grade pentane was carried out.

...../2

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL AND FILTERS

DATE January 21, 1980

PROJECT NO. 24769 C

A subsequent rinse with pentane of all interior and connecting surfaces was carried out and collected in a 600 ml Kuderna Danish concentrator. Similarly, pentane rinsings from other components of the train which were cleaned by different procedures were combined in the associate Kuderna Danish concentrator.

B) Cleaning of Glass Probes

Because of their extreme length it was not possible to heat the probes in an oven.

Some probes required soaking of the ball end in methylene chloride to remove visible contamination. Probes were cleaned by generous rinsing of the interior and connecting surfaces with methylene chloride (2X), acetone (2X) followed by hexane (2X). When the probes were rinsed they were held at approximately a 30°angle to the horizontal and rotated about their long axis. This procedure provides an extended contact time and complete contact of the interior surfaces.

Visual examination of the glass probe both of the exterior and interior surfaces was necessary. Repeated rinsings were required to remove particulate visible when looking into the interior of the probe along its long axis.

Glass probes were rinsed once more with pentane prior to collecting a final pentane rinse in the associated Kuderna Danish concentrator.

C) Cleaning of Frittered Glass Filter Support Disc

Because of the nature of the frittered glass filter support disc and its thickness it was decided not to expose it to the detergent soak.

The discs were soaked in acetone for 12 hours, generously rinsed with acetone then transferred to a soak in hexane for 12 hours followed by generous rinsing. The discs were then transferred to a 250°C forced air oven for a minimum of 12 hours. After removal from the oven the discs were allowed to cool on the paper covered laboratory bench and then generously rinsed with pentane to waste.

A final generous rinse with pentane sufficient to completely wet the frittered material was carried out while the disc was held above the Kuderna Danish concentrator and rotated.

...../3

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL AND FILTERS

DATE January 21, 1980

PROJECT NO. 24769 C

D) Nickel plated stainless steel nozzles and connectors were soaked in Decon 75 water overnight. They were thoroughly scrubbed with soft bristle brushes both inside and out. Nozzles were then rinsed with tap water followed by a once distilled water rinse. A generous solvent rinsing; methylene chloride 2X, acetone 2X followed by hexane 2X was followed by a pipe cleaner check of internal cleanliness

The absence of discolouration such as grease or oil on the pipe cleaner was considered as sufficient evidence of internal "visual" cleanliness.

Nozzles and connectors were then held for a minimum of 12 hours in a 250°C forced air oven. After removal from the oven nozzles and connectors were allowed to cool on the paper covered laboratory bench. After cooling the interior and connecting surfaces were rinsed with pentane IX to waste.

A final rinse of the internal and connecting surfaces with pentane IX was collected in the associated Kuderna Danish concentrator.

E) Cleaning of Aluminum Foil and Sealing of train Components.

Commercially available heavy duty aluminum foil was cleaned up by the following procedure. Sheets of foil 24" x 16" were soaked in Decon 75 - water solution for a minimum 24 hours then rinsed with tap water followed by a rinse with IX distilled water.

The sheets were then immersed in acetone for 3 hours allowed to drain then immersed in hexane for 3 hours and allowed to drain. The foil sheets were then allowed to air dry prior to being placed in a 250°C forced air oven for a minimum of 12 hours.

The aluminum foil was proven as a batch by rinsing both sides of 5 to 6 representative sheets with pentane which was collected in a Kuderna Danish concentrator, concentrated and assayed by Gas Liquid Chromatography (GLC).

The proven aluminum foil was employed to seal off the interior and connecting surfaces of all glassware and metal components previously cleaned. Components were not assembled into the train at Nucro-Technics but sufficient components to constitute a train were cleaned, proven together, sealed with the aluminum foil and provided to Envirocon (Eastern) Limited as a set.

..../4

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL AND FILTERS

DATE January 12, 1980

PROJECT NO. 24769 C

F) Cleaning and Proving of Glass Fiber Filters

Glass fiber filters of a nominal 5 inch diameter were provided by Envirocon (Eastern) Limited.

The filters were cleaned by soxhlet extraction with pentane. Two soxhlet apparatus were operated containing three filters each for 9 hours at a cycle rate of 3 per hour. After initial soxhlet extraction the charge of pentane was replaced with fresh pentane and a second extraction cycle for 8 hours at a cycle rate of 3 cycles per hour was conducted. The initial pentane charge (~200 mls) and two (~30 ml) rinses of the receiver from both soxhlets was combined and quantitatively transferred to a Kudern Danish concentrator for concentration and subsequent GLC assay.

After extraction filters were dryed at 120°C for a minimum 12 hours and preweighed. Each filter was numbered,sealed in precleaned aluminum foil and assigned to a train.

G) Cleaning Preparation and Proving of Florisil Cartridges

The glass cartridges received solvent rinsing only of methylene chloride IX, acetone 2X, hexane 2X. After air drying the glass cartridges were transferred to a 250°C forced air oven for a minimum of 12 hours.

Glass wool was cleaned by serial extraction with pentane in a beaker. After pouring off the excess pentane, the beaker containing the glass wool was placed at the rear of a fume hood and allowed to dry. When dry the beaker and glass wool was transferred to a 120°C oven for storage.

A portion of the glass wool cleaned and stored as above was extracted by shaking in pentane for five minutes. The pentane extract was collected in a Kuderna Danish concentrator for concentration and subsequent GLC assay.

Florisil 30/60 mesh was cleaned in a vertical tube furnace as a batch. The process employed heats the material to 650°C over a 12 hour period after which it is held at 650°C for a minimum 12 hours and then allowed to cool for an additional 12 hours. During the heating process florisil filtered air under pressure purges the quartz column. After removal from the tube furnace the florisil is held in a 120°C oven until use.

...../5

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM AND FILTERS

DATE January 21, 1980

PROJECT NO. 247629 C

Individual cartridges were filled with florisil prepared as above. A layer of glass wool prepared as above is tamped in place with a glass rod over the florisil.

Two previously packed and proven cartridges were submitted by Envirocon (Eastern) Limited. Both cartridges underwent the same cleanup and proving procedures for newly manufactured cartridges.

Each cartridge was eluted with 2x100 ml portions of 10% methylene chloride in pentane and allowed to drain. This was followed by 2x100 ml portions of pentane and allowed to drain. A final eluate of 100 ml pentane was collected in individual Kuderna Danish concentrators for each cartridge. The eluate was concentrated and analyzed by GLC.

In review, the components of the florisil cartridge were cleaned up individually, then assembled or packaged with a final cleaning procedure of the assembly followed by an individual proving of each florisil cartridge.

After elution with pentane, the individual cartridges were tagged with an identification number and then, blown down with florisil filtered air. Cartridges were reactivated by a 24 hour holding period in a 120°C oven. After removal from the oven the ends of the florisil cartridge were sealed with a double layer of proven aluminum foil.

Concentration of Pentane for GLC Assay

Pentane from a proving rinse or extraction was collected and transferred to a Kuderna Danish concentrator. A keeper of 1.0 milliliter iso-octane and two or three precleaned boiling chips were added.

Samples were concentrated to approximately 5 milliliters employing the main body of the Kuderna Danish concentrator and a three ball macro Snyder column and water bath at 60°C.

The main body of the Kuderna Danish was removed and a micro Snyder column substituted for the macro Snyder. The concentration was continued on a heating apparatus to 1.0 milliliter or less, then made up to 1.0 milliliter.

The iso-octane concentrate was transferred to precleaned glass micro vials with aluminum foil lined screw caps for holding until GLC assay.

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL AND FILTERS

DATE January 21, 1980

PROJECT NO. 24769 C

Results of GLC Assay

Table 1 - Trains for 2nd Pre Burn Stack Sampling.

| Part | Concentration PCB ng/ml | Total PCB in train ng |
|---|---|---|
| Train 1 | $\leq$ 2.67 | $\leq$ 2.67 |
| Train 2 | $\leq$ 1.33 | $\leq$ 1.33 |
| Train 3 | $\leq$ 19.12 | $\leq$ 19.12 |
| Train 4 | $\leq$ 37.44 | $\leq$ 37.44 |
| Train 1 of Dec. 28 | $\leq$ 25.98 | $\leq$ 25.98 |
| Train 1 Supplemental | None Detected | None Detected |

...../7

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL AND FILTERS

DATE January 21, 1980

PROJECT NO. 24769 C

Table 2 - Florisil Cartridges for 2nd Pre Burn Stack Sampling.

| Part # | Concentration PCB ng/ml | Issued | Total PCB in cartridge ng |
|---|---|---|---|
| Tag #1 Cartridge #11 - 1st Time | ≤ 3.33 | Yes | ≤ 3.33 |
| Repeat Cleanup #11 | ≤ 2.59 | Yes | ≤ 2.59 |
| Tag #2 Cartridge #10 not issued | ≤ 1338 | No | ≤ 1338 |
| #3 | ≤ 0.72 | Yes | ≤ 0.72 |
| #4 | ≤ 0.87 | Yes | ≤ 0.87 |
| #5 not issued | ≤ 2597 | No | ≤ 2597 |
| #6 | ≤ 51.49 | Yes | ≤ 51.49 |
| #7 | ≤ 4.61 | Yes | ≤ 4.61 |
| #8 | ≤ 9.01 | Yes | ≤ 9.01 |
| #9 | ≤ 34.47 | Yes | ≤ 34.47 |
| #10 | ≤ 30.38 | Yes | ≤ 30.38 |

...../8

SUBJECT CLEANING AND PROVING OF STACK SAMPLING TRAINS, FLORISIL CARTRIDGES, ALUMINUM FOIL AND FILTERS DATE January 21, 1980

PROJECT NO. 24769 C

Table 3 - Ancillary Supplies for 2nd Pre Burn Stack Sampling

| Part | Concentration PCB in ng/ml | Amount Total PCB ng |
|---|---|---|
| Glass Fiber Filter | 130 | 130 (for 6) |
| Florisil 30 (60 mesh) | 49.50 | 49.50 |
| Glass Wool | 1.13 | 1.13 |
| Aluminum Foil | None Detected | None Detected 3 pieces |
| Aluminum Foil | 7.44 | 7.44 6 pieces |

The results of GLC assay for the 5 trains, florisil cartridges and ancillary supplies are presented in tabular format, tables 1, 2 and 3.

Fred S. Seymour, B.Sc.
Director of Technical Marketing.

NUCRO TECHNICS LIMITED

ANALYTICAL REPORT

2000 ELLESMERE ROAD, UNIT 16, SCARBOROUGH, ONTARIO M1H 2W4 416/438-6727

DATE January 21, 1980

PROJECT NO. 24769 E

John Trought, P. Eng.
Manager
Envirocon (Eastern) Limited
P.O. Box 1339
Downsview, Ontario
M3H 5W3

SUBJECT EXTRACTION OF THREE STACK SAMPLING TRAINS, FROM SECOND PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

Procedures :

A) Disassembly of Trains

Stack sampling trains were transported intact as assembled for the stack testing by Envirocon (Eastern) Limited (EEL) personnel to Nucro-Technics Limited. The intake and outlet openings were sealed after the completion of the on stack portion of the test by EEL personnel. Precleaned and proven aluminum foil provided by Nucro-Technics Limited (NTL) was employed for the sealing of the trains.

Trains were partially disassembled prior to extraction. As each section was removed from the train the inlet and outlet openings were sealed with aluminum foil. Seals were secured with light metal wire twisted tight. Additionally each major component of the train was labelled with a tag as to component number and train. All components with the exception of glass probes were stored together until extraction.

B) Sample Recovery and Extraction

(i) Glass sampling probe, nozzle, cyclone bypass and front section of filter holder.

Shotgun brushes, all metallic, with bristles of brass in 0.410 inch boresize were previously found to be suitable. Cleaning and proving procedures were covered under the material section of report #24769 D.

The shotgun brushes were employed with braided stainless steel wire as a pull through. The wire was precleaned by solvent washing in methylene chloride (IX), acetone (2X) and hexane (2X). Two passes of the brush were found to be necessary. Later the brush was rinsed with pentane. The interior surfaces of the probe were rinsed with successive washes of pentane while the probe was slowly rotated.

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SUBJECT EXTRACTION OF THREE STACK SAMPLING TRAINS, FROM SECOND PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 21,1980

PROJECT NO. 24769 E

The pentane washes and recovered particulate were collected in a previously cleaned and proven glass container.

The interior surfaces of the nozzle, connector, cyclone bypass and front filter holder were generously rinsed (four rinses) with pentane. The pentane rinses and recovered particulate were collected in a previously cleaned and proven container.

(ii) Glass Fiber Filters

After removal from the filter holder the filters went directly to soxhlet extraction. The initial charge of pentane was approximately 200 ml and the filter and residue was extracted in a precleaned and batch proven thimble for a minimum of eight hours at a cycle rate of three per hour. The initial pentane charge and two (∿30 ml) pentane rinses of the receiver were combined in a previously cleaned and proven glass container. See Table 1 - Filter Weight.

(iii) Frittered Glass Filter Support and Rear Filter Holder

The frit was extracted by soaking in pentane in a foil covered beaker overnight. Two additional rinses with pentane sufficient to soak the frit were combined with the first extract and the total extract was transferred to a previously cleaned and proven glass container.

The interior surfaces of the rear half of the filter holder were rinsed three times and the rinses combined with the frit extract.

(iv) Impinger Connecting Glassware

Impinger connecting glassware with visible water droplets was rinsed with two one ml portions of pesticide grade acetone per piece. Four rinses with pentane of the interior surfaces of each piece were conducted and combined with the acetone in a precleaned proven glass container. A final rinse with pentane of each piece was collected in a separate precleaned and proven glass container.

(v) Impinger Contents

Water for impinger pre-loading was supplied by the Ministry of the Environment. Pre-sampling water volume in impingers 1 and 2 was determined by EEL personnel.

..../3

SUBJECT EXTRACTION OF THREE STACK SAMPLING TRAINS, FROM SECOND PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 21, 1980

PROJECT NO. 24769 E

Individual impingers were not preweighed and labelled. After receipt each impinger was labelled as to train and impinger sequence number. Prior to extraction the total weight of each impinger and contents was determined by NTL personnel. See Table 2 - Impinger Contents.

The total water content of all impingers was equally distributed between all of the impingers of a train. A nominal 100 ml of pentane was added to each impinger and the impinger and contents extracted by vigorous shaking for a minimum of five minutes. After shaking the phases were allowed to separate and the pentane (upper phase) was drawn off by aspiration employing a long tipped 25 or 50 ml pipette and a rubber bulb. The extraction was repeated four times with each extraction being successively filtered through anhydrous sodium sulfate retained in a proven glass funnel with proven glass wool. The impinger pentane extracts for a train were combined with the impinger connecting glass ware extract.

In some cases the total volume of pentane extract of the impinger exceded one litre and an additional proven glass container was necessary.

After extraction the remaining contents of the impinger (water) was discarded. The impingers were allowed to air dry and reweighed. See Table 2 - Impinger Contents

(vi) Extraction of Florisil Cartridges

The entire contents of the cartridge including glass wool was expelled into a precleaned and batch proven cellulose thimble. The florisil and glass wool was extracted in a Soxhlet extraction apparatus for a minimum of eight hours at a cycle rate of three per hour. The initial pentane charge of ∿200 ml and two ∿30 ml rinses of the receiver were combined in a cleaned and proven glass container.

C) Materials

Solvents - Pesticide Grade, Glass Distilled, Pentane, Acetone, Methylene Chloride and Hexane were obtained from Caledon Laboratories.

Glass Wool - previously cleaned and proven. See Report #24769 C.

Anhydrous Sodium Sulfate:
Anhydrous sodium sulfate ACS grade (Canlab) was cleaned and proven as previously described. See report #24769 D.

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SUBJECT EXTRACTION OF THREE STACK SAMPLING TRAINS, FROM SECOND PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 21, 1980

PROJECT NO. 24769 E

D) Cleaning and Proving of Glassware and Final Containers for Extracts

Glassware and glass containers for the pentane extracts were cleaned by. an extensive cleaning process. A minimum 12 hour soak in Decon 75 water was followed by generous rinsing with tap water, filtered by distilled water. Solvent rinsings in sequence were methylene chloride 1X, acetone 2X and hexane 2X. After air drying glassware was baked in a 250°C forced air oven for a minimum of 12 hours. After cooling a rinse with pentane to waste was conducted.

A final rinse with pentane ~15 ml per piece or more was collected and concentrated for GLC assay.

Cellulose thimbles for Soxhlet extraction were cleaned up by Soxhlet extraction as previously described in report #24769 D. In addition to the previous batch approval by GLC assay an additional three thimbles from the top, middle and bottom of the load were each extracted in a Soxhlet apparatus with pentane for 8 hours at a cycle rate of three per hour. The extracts were combined and concentrated for GLC assay. The results of the GLC assay for each of the three thimbles are reported in Table 3 - Total PCB content of Materials and Glassware Employed in Sample Extraction.

Fred Seymour, B.Sc.
Director of Technical Marketing.

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SUBJECT EXTRACTION OF THREE STACK SAMPLING TRAINS, FROM SECOND PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 21, 1980

PROJECT NO. 24769 E

TABLE 1 - FILTER WEIGHTS

| Sample Designation | Final Weight | Initial Weight | Residue Weight |
|---|---|---|---|
| Train #1, Kiln | 1.0220g | 0.8130g (from train #4) | 0.2090g |
| Train #2, Kiln | 0.9394g | 0.8037g | 0.1357g |
| Standby Train | 0.8047g | 0.8002g | 0.0045g |
| Lab Run | _____ | _____ | _____ |

...../6

SUBJECT EXTRACTION OF THREE STACK SAMPLING TRAINS, FROM SECOND PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 21, 1980

PROJECT NO. 24769 E

TABLE 2 - IMPINGER CONTENTS

| IMPINGER # & TRAIN | FINAL WEIGHT | EMPTY WEIGHT | NET WATER GAIN |
|---|---|---|---|
| Train #1, Kiln | | | |
| 1 | 768g | 515.5g | 252.5g |
| 2 | 740.5 | 470.4 | 270.1 |
| 3 | 735.9 | 459.1 | 276.8 |
| 4 | 621.8 | 506.4 | 115.4 |
| 5 | 520.4 | 496.1 | 24.3 |
| | | TOTAL | 939.1g |
| Train #2, Kiln | | | |
| 1 | 749.3g | 517.2g | 232.1g |
| 2 | 750.8 | 470.9 | 279.9 |
| 3 | 704.5 | 471.7 | 232.8 |
| 4 | 686.1 | 499.0 | 187.1 |
| 5 | 478.1 | 476.1 | 2.0 |
| | | TOTAL | 933.9g |
| Standby Train | | | |
| 1 | 615.9g | 520.7g | 95.2g |
| 2 | 619.1 | 521.6 | 97.5 |
| 3 | 475.3 | 475.3 | |
| 4 | 473.5 | 473.5 | |
| 5 | 474.5 | 474.5 | |
| | | TOTAL | 192.7g |

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SUBJECT EXTRACTION OF THREE STACK SAMPLING TRAINS, FROM SECOND PRE BURN TRIALS AT ST. LAWRENCE CEMENT, MISSISSAUGA

DATE January 21, 1980

PROJECT NO. 24769 E

TABLE 3 - TOTAL PCB CONTENT OF MATERIALS AND GLASSWARE EMPLOYED IN SAMPLE EXTRACTION

| PART | CONCENTRATION TOTAL PCB ng/ml | TOTAL PCB in PART ng |
|---|---|---|
| MOE GLASSWARE | | |
| Batch 1, 12-8oz. | 9.42 | 9.42 |
| Batch 2, 12-8oz. | 6.97 | 6.97 |
| Batch 3, 12-8oz. | 14.1 | 14.1 |
| Batch 4, 12-8oz. | 77.2 | 77.2 |
| Batch 1, 12-32 oz. to EEL | 0.33 | 0.33 |
| Batch 1, 12-32 oz. NTL | 4,832 Rejected | 4,832 Rejected |
| Batch 1, Recycled | 39.14 | 39.14 |
| Batch 2, NTL | 1.09 | 1.09 |
| Beakers, Funnels & Pipettes | 16.9 | 16.9 |
| Batch 4, NTL | 3.12 | 3.12 |
| Thimble, Top Layer | 2.83 | 2.83 |
| Thimble, Middle Layer | 0.18 | 0.18 |
| Thimble, Bottom Layer | 2.21 | 2.21 |

# APPENDIX 3

NOTE

For results reported, the following conditions apply:

1. Results for isomer identification and quantitation were based on the 27 PCB isomer mix used in this laboratory.

2. No allowance was made for peaks that were present in the samples but did not correspond to the retention times of known isomers.

3. The values reported for the perchlorinated samples have not been corrected for biphenyl content.

4. The decachlorobiphenyl/PCB conversion factor was calculated using only the PCB isomers that were identified in the samples based on the 27 isomer standard mix.

## Analysis for PCB Isomers in Extracts from Stack Gas and Process Materials from St. Lawrence Cement Kiln

Pentane extracts of stack and process samples taken at the St. Lawrence Cement plant in Mississauga were received from the processing laboratory (Nucrotechnics Ltd.) (100 - 1500 mls).

The pentane extracts were evaporated to approximately 8 mls using a Kuderna Danish evaporator (40 minutes). The extract was further reduced to 4 mls using a micro Snyder Condenser. These steps have been tested and optimised to minimize losses of volatile PCB components.

A 1 ml aliquot of the extract was tested by shaking with metallic mercury to remove sulphur-containing materials. These compounds must be removed:

a) To avoid spurious "false positive" peaks and base line interferences in gas chromatographic analyses of PCB's.

b) To avoid serious interferences with the "perchlorination" method.

The extract was "cleaned up" using a 24 cm x 0.6 cm chromatographic column, packed with 1% water-deactivated Florisil. PCB's were eluted with pentane, whilst most interfering organics and pesticides were retained by the Florisil.

The amount of eluting solvent is previously determined by eluting a PCB isomer mixture from a similar column with pentane (approximately 16 mls) and determining the pentane volume needed to recover all PCB components. The eluate was evaporated to 4 mls, using a micro-Snyder distillation apparatus, and then divided into 2 portions. One portion is used for PCB isomer analysis, and the other for perchlorination.

### PCB Isomer Analysis by Electron Capture Gas Chromatography

The extract was analysed on an electron capture detector equipped gas chromatograph at the following conditions: The PCB isomer analysis was performed using a

3.5 m x 0.2 cm Pyrex column packed with 1% [illegible] Chromosorb W. The chromatographic conditions were: Injector 250°C, Detector 300°C, Column programmed 160 - 220°C at 1°/min., 8 minutes hold at 220°C. Gas flow 18 mls/min, Argon/5% methane.

PCB components in the resulting chromatogram were identified and quantitated by comparison with a known PCB isomer standard mixture, containing 27 isomers ranging from mono - hexa-chlorobiphenyls. The composition of the mixture was chosen to reflect:

1) Major components ( 1%) present in commercial Aroclors.

2) Compounds which were well resolved under the state of chromatographic conditions.

3) Isomers at present commercially available.

Instrument control data acquisition and processing was carried out using a Hewlett Packard 3353C chromatography data system. Some compounds appear in the chromatogram with the same retention times as components in commercial Aroclors, but for which corresponding single isomers could not be included in the mixture. These were quantitated using response factors previously derived from the "fingerprint" of the Aroclor in which they occur. PCB results were calculated and reported as:

Sum of all monochlorobiphenyls
Sum of all dichlorobiphenyls
Sum of all trichlorobiphenyls
Sum of all tetrachlorobiphenyls
Sum of all pentachlorobiphenyls
Sum of all hexachlorobiphenyls

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Total PCB's

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The above report is necessary to calculate an average level of chlorination of the compounds present for inclusion in the next stage of analysis. Report will indicate:

e.g. Total PCB's - 29 ng/m$^3$ (average % Cl = 25)

Whilst we consider the method as a considerable improvement on current PCB quantitation methods, it still leaves some problems on precise identification of all PCB isomers present. Development work is ongoing to introduce the use of capillary column gas chromatography to enhance separation, improve accuracy of identification and to improve sensitivity.

Perchlorination

As a quantitative cross-check, selected samples were reacted with antimony pentachloride to convert all PCB compounds to one compound - decachlorobiphenyl. After quantitation, the PCB value obtained was compared with that for the same analysis.

A 5 ul aliquot of the perchlorination sample was analysed for biphenyl. If biphenyl was detected in the sample, the results obtained from the perchlorination reaction have to be corrected for the presence of biphenyl in the sample. The extract was analysed on a Photoionization detector (10.2 ev Lamp) equipped gas chromatograph at the following conditions: Column - 6' x 1/8" Nickel packed with 3% Dexsil 300 GC on Chromosorb WHP, Detector 300°C, Injector 210°C, Column 240°C, Gas flow 40 mls/min.

The perchlorination portion of the extract was transferred to a specially designed reaction tube and azeotropically distilled with 3 x 1 ml aliquots of chloroform to remove pentane, which interferes with the reaction.

The chloroform extract was evaporated to 0.3 ml prior to addition of 0.2 mls of the perchlorination reagent, i.e. antimony pentachloride.

The reaction tube was sealed, and the mixture was heated at 160°C for 15 hours, in a special reaction block, and the reaction terminated by the addition of hydrochloric acid.

The sample was extracted with hexane (5 times) and the resulting extract cleaned up on a mixed Alumina/Florisil column. Decachlorobiphenyl was eluted with 10 mls hexane.

The eluate from the column was diluted to a suitable volume for GC analysis of decachlorobiphenyl. Gas chromatographic analysis was performed by electron capture detection, using a 6' x 2 mm ID column packed with 1% Dexsil 400 on

Ultrabond II. The chromatographic conditions were: Injector 250°C, Electron Capture Detector 300°C, Column 240°C, Gas flow 40 mls/minute Nitrogen.

The resulting chromatogram was compared to a standard curve generated by injection of known amounts of decachlorobiphenyl standard.

From the amount of decachlorobiphenyl detected, the total amount of PCB was determined by application of a conversion factor.

The calculation of the conversion factor was based on the total ng's of the PCB isomers detected in the isomer analysis and an estimate of the average chlorine content of the PCB isomers detected in the isomer analysis segment of the analysis.

DISCUSSION:

Isomer Analysis:

Analysis of PCBs in the stack samples and process samples was carried out by "best available" packed column gas chromatography. Due to the limit in capability of this system to separate individual PCB isomer, coupled with limited availability of pair PCB isomers for standards, only 27 isomers could be used to calibrate the system and as quantitation standards. As a result, any PCB components present in the sample extracts, but not corresponding in retention time to one of these 27 isomers were not included in the analytical quantitation. In general, examination of the chromatograms from these samples showed that at least 70% of the peaks present were quantitated against PCB standards.

Whilst a fairly broad range of cleanup procedures were used to remove interfering, non-PCB compounds, each of precise information on all compounds present in the stack gases and process materials make it impossible to state that all non-PCB components were removed. Electron capture detection is not specific for PCB isomers thus the possibility exists that some peaks measured as PCBs were interfering compounds. Due to poorer rsolving power of packed columns vs. capillary columns, it is probable that such false positive results would be more significant in packed column operation than in capillary column operation. Further analytical development work is underway to identify other stack gas components (GC-MS) to improve cleanup procedures.

Gas Chromatography - Mass Spectrometry: (GC-MS)

Qualitative examination of selected stack gas samples by capillary GC-MS has been undertaken to attempt to speciate PCB components present. This work has indicated the presence of a wide range of other materials (mainly hydrocarbons) which at present mask any PCB components present. High pressure liquid chromatographic cleanup is now being evaluated for removal of these hydrocarbons.

Perchlorination:

If samples can be adequately cleaned up to remove hydrocarbons and sulfur compounds, the technique of perchlorination as a method of qualitating PCBs in stack and process samples is practical. PCB isomers present in the sample are converted to a single unique derivative - decachlorobiphenyl, which can be positively identified and accurately quantitated. After correction for any biphenyl present in the samples (measured separately) it can be stated from theoretical considerations that the total amount of PCBs present in the sample, taking into account isomer distribution in stack gases, is between 40% - 70% of the decachlorobiphenyl value. Such an approach avoids inaccurate results caused by non-identification of PCB components, or false positives from inter-ferences, but does limit the data on actual PCB species present.

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## LABORATORIES

EXPRESS ADDRESS:
RESOU ES ROAD
HIGHW. 401 & ISLI GTON AVE.
TORONTO, ONTARIO

Municipality: .....
Source: ..... Region :
Program: .....
Date sampled: ..... by: .....
Date analysed: ..... Date reported: .....

Report to: V [illegible]
Address: [illegible]

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|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT | DAY | TIME |
|---|---|---|---|---|---|---|
| Pg 3612 | | Train #6 Blank | 1st, 2nd, 3rd pentane Rinse of Probe Nozzle, front half of filter | holder | ✓ | — |
| Pg 3613 | | ✓ ✓ ✓ ✓ ✓ | ✓ of Rear half of filter holder | | ✓ | ✓ |
| Pg 3614 | | ✓ ✓ ✓ | Florisil Cartridge #13 SOXHLET EXTRACTION | | — | ✓ |
| Pg 3615 | | ✓ ✓ ✓ | 4th Rinsing of rear filter holder | | ✓ | ✓ |
| Pg 3616 | | ✓ ✓ ✓ | Probe Nozzle, front half of filter holder | | ✓ | ✓ |
| Pg 3617 | | ✓ ✓ ✓ | Blank train fiberglass filters - SOXHLET EXTRACTION | | — | ✓ |
| | | | | | | |
| | | | | | | |

| LAB. NUMBER | | PCB's Total mg | Aroclor 1242/1260 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| [illegible] 3612 | | | | | | | | | | | |
| 13 | | | | | | | | | | | |
| 14 | | X | | | | | | | | | |
| 15 | | | | | | | | | | | |
| 16 | | | | | | | | | | | |
| 17 | | X | | | | | | | | | |
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MOL 0951

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TORONTO, ONTARIO

Municipality: .....
Source: ..... Region: .....
Program: .....
Date sampled: ..... by: .....
Date analysed: ..... Date reported: .....

Report to: .....
Address: .....

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | *CHECK BELOW IF CHLORINE PRESENT | DIS | TOT |
|---|---|---|---|---|---|---|
| [illegible] 3618 | | Clinker Cooler C Drain #7 4th pentane rinse, probe nozzle, front half of filter | | | ✓ | — |
| [illegible] 3619 | | ✓ ✓ ✓ ✓ 1st, 2nd, 3rd ✓ ✓ ✓ ✓ ✓ ✓ ✓ ✓ ✓ holder. | | | ✓ | — |
| [illegible] 3680 | | ✓ ✓ ✓ ✓ 4th rinsing of Rear filter holder. | | | ✓ | — |
| [illegible] 3681 | | ✓ ✓ ✓ ✓ Cartridge #3 | | | — | ✓ |
| [illegible] 3682 | | ✓ ✓ ✓ ✓ 4th rinsing 4 pieces impingers connecting glassware | | | ✓ | — |
| [illegible] 3683 | | ✓ ✓ ✓ ✓ First 3 rinsings of Rear filter holder. | | | ✓ | — |
| [illegible] 3684 | | ✓ ✓ ✓ ✓ Pentane Rinse Probe, filter. | | | — | ✓ |
| | | | | | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | FOR [illegible] | [illegible] | [illegible] | 29/1980 | | | | | | |
| | | | | | | | | | | | |
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| | | | | | | | | | | | |
| | | ✓ | | | | | | | | | |
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| | | X | | | | | | | | | |
| | | | | | | | | | | | |

MOE 1051

| Sample No. | Total ng. PCB isomer | Average %Cl | Total ng. DCBP | Corrected Total ng. PCB |
|---|---|---|---|---|
| [illegible]-3674 | 257.7. | 0.46 | [illegible] | 1[illegible] |
| | | | | |
| [illegible]-3677 | 197.6 | 0.49. | 93.7 | 48.4 |
| | | | | |
| [illegible]-3691 | 318.5 | 37.2 | 1904 | 424 |
| | | | | |
| [illegible]-3684 | 240.0 | 35.9 | 816. | 103 |
| | | | | |
| | | | | |
| [illegible]-3687 | 4771 | 39.3 | 5228 | 4110. |
| | | | | |
| | | | | |
| [illegible]-3688. | 405. | 34.5 | 154 | 71. |
| | | | | |
| | | | | |
| [illegible]-3698 | 453 | 33.4 | 280 | 139 |
| | | | | |
| | | | | |
| [illegible]-3699 | 4933. | 27.3 | 25,600 | 10,500 |
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Municipality: [illegible]
Source: [illegible] Region: [illegible]
Program: [illegible]
Date sampled: Jan 3 80 by: [illegible]
Date analysed: [illegible] Date reported: Jan 7, 80

Report to: [illegible]
Address: [illegible]

C
Bi
Ba

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | √CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| From | | From #2 a Stock Sample — liquid-liquid | | |
| | | extraction from 5 impingers & connections | | |
| 0-103 | | rinse with tap water & acetone. | | |
| | | - two aliquots (2) | | |
| | | - extracted Jan 7, 80 - successively filtered through [illegible] | | |
| | | - 2 fractions collected. Sodium [illegible] | | |
| | | Fractions Spiked with Alkyl chlorides Batch # 2-32 | | |
| | | | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 0-104 | [illegible] | | | | | | | | | | |
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MOE 0151

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RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: [illegible]
Source: [illegible] Region:
Program:
Date sampled: June 3 80 by: Investigation
Date analysed: Date reported:
Sampled June 3, 80 MTC

Report to: [illegible]
Address:

C
Bi
Ba

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| | | Influent extract of impingers - Standing Tem. | | |
| | | - Impinger - Liquid Extraction with final rinse DCM | | |
| 80-101 | | all combined. | | |
| | | - MB Cartridge present - Ostensibly | | |
| | | - Excess of DCM rinse in the Impinger | | |
| | | | | |
| | | Containing some of Metal Glass Beads | | |
| | | | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | |
| 80-101 | MB | | | | | | | | | | |
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MOE 0514

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EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: [illegible] Region
Source: [illegible] Region;
Program:
Date sampled: June 3 80 by: Environment
Date analysed: June 3 80 Date reported: [illegible]

Report to: [illegible]
Address:

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| | | Inhibit [illegible] of plankton [illegible] | | |
| | | from Trent Aire a stagnant | | |
| P-102 | | - chemical [illegible] | | |
| | | effluents 3/week + 2 [illegible] | | |
| | | added. | | |
| | | - Conduit mixing [illegible] | | |
| | | | | |
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| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | |
| P-102 | [illegible] | | | | | | | | | | |
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MOE 0-151

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: [illegible] Township
Source: [illegible] Region:
Program:
Date sampled: [illegible] by: [illegible]
Date analysed: [illegible] Date reported: [illegible]

Report to: [illegible]
Address:

C
Bi
Ba

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| [illegible] | | [illegible] | | |
| | | [illegible] | | |
| [illegible] | | [illegible] | | |
| | | [illegible] | | |
| | | | | |
| | | [illegible] | | |
| | | | | |
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| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | |
| [illegible] | [illegible] | | | | | | | | | | |
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MOE 0951

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: [illegible] Conservation
Source: [illegible] Region
Program:
Date sampled: June 3 80 by: Environ
Date analysed: Date reported:

Report to: [illegible]
Address:

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| | | Sandfly Train - Water Start Sample. | | |
| | | Effluent Station 1 Filter (one) Creek. | | |
| | | Equitone Mix 1m & Bur (2) apart onto | | |
| 80-104 | | 3/4 Hose, 1 2x30 in Line of Station | | |
| | | | | |
| | | Container filling to MOE Glass with Sample | | |
| | | | | |
| | | | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | |
| 80-104 | | | | | | | | | | | |
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MOE 0021

Sample no. P20-122

Location ST LAWRENCE

TRAIN 1 @ STAGE SAMPLE

IMPINGERS

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 1 | 326.7 | | | | | | | |
| 26 | 60.6 | | | | | | | |
| 22' | | | | | | | | |
| 25 | 73.8 | | | | | | | |
| 23 | 83.8 | | | | | | | |
| 246 | | | | | | | | |
| 225 | 27.6 | | | | | | | |
| 44' | | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 80.9 | | | | | | | |
| 2256' | 63.2 | | | | | | | |
| 2346 | | | | | | | | |
| 2255' | 160.8 | | | | | | | |
| 2235' | 78.7 | | | | | | | |
| 2246 | | | | | | | | |
| 2233' | 46.4 | | | | | | | |
| 2345 | 151.1 | | | | | | | |
| 22455' | 97.9 | | | | | | | |
| 2356 | | | | | | | | |
| 2233'66' | 0.6 | | | | | | | |
| 33'44' | 17.7 | | | | | | | |
| 2244'55' | | | | | | | | |
| 22344'5 | | | | | | | | |
| 23455'6 | | | | | | | | |
| 2233'44' | | | | | | | | |
| 233'44'5 | | | | | | | | |
| Total | | | | | | | | |

1269

Sample no. P80-101

Location ST LAWRENCE CEMENT PLANT
STANDBY TRAILER

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 1 | 143 | | | | | | | |
| 26 | | | | | | | | |
| 22' | 200 | | | | | | | |
| 25 | 0.7 | | | | | | | |
| 23 | | | | | | | | |
| 246 | 0.2 | | | | | | | |
| 225 | | | | | | | | |
| 44' | 1.6 | | | | | | | |
| 22'46 | 0.4 | | | | | | | |
| 245 | | | | | | | | |
| 2256' | 0.4 | | | | | | | |
| 2346 | | | | | | | | |
| 22'55' | | | | | | | | |
| 22'35' | | | | | | | | |
| 22'446 | | | | | | | | |
| 22'33' | | | | | | | | |
| 23'45 | | | | | | | | |
| 22'455' | 0.3 | | | | | | | |
| 23456 | | | | | | | | |
| 22'33'66' | 0.5 | | | | | | | |
| 33'44' | | | | | | | | |
| 22'44'55' | | | | | | | | |
| 22'344'5 | | | | | | | | |
| 22'3455'6 | | | | | | | | |
| 22'33'44' | | | | | | | | |
| 233'44'5 | | | | | | | | |
| Total | 38.4 | | | | | | | |

Samp'e no. 239-102

Location ST LAWRENCE CEMENT PLANT
TRAIN 1 @ STACK
GLASS FIBRE FILTER / CANS

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 1 | 22.8 | | | | | | | |
| 26 | | | | | | | | |
| 22' | | | | | | | | |
| 25 | 0.9 | | | | | | | |
| 23 | 1.4 | | | | | | | |
| 246 | 0.9 | | | | | | | |
| 225 | | | | | | | | |
| 44' | | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 0.4 | | | | | | | |
| 2256' | 2.0 | | | | | | | |
| 2346 | | | | | | | | |
| 2255' | | | | | | | | |
| 2235' | 0.3 | | | | | | | |
| 22446 | | | | | | | | |
| 2233' | | | | | | | | |
| 2345 | | | | | | | | |
| 22455' | 1.1 | | | | | | | |
| 23456 | 0.1 | | | | | | | |
| 2233'66' | 1.1 | | | | | | | |
| 33'44' | 1.9 | | | | | | | |
| 2244'55' | | | | | | | | |
| 22344'5 | 0.2 | | | | | | | |
| 223455'6 | | | | | | | | |
| 2233'44' | | | | | | | | |
| 2334'45 | | | | | | | | |
| Total | 33.1 | | | | | | | |

Sample no. P10-103

Location ST LAWRENCE

GLASS FIBRE FILTER

TAKEN @ [illegible]

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 4.1 | | | | | | | |
| | | | | | | | | |
| 26 | | | | | | | | |
| 22' | | | | | | | | |
| 25 | 20.3 | | | | | | | |
| 23 | 0.2 | | | | | | | |
| 246 | 0.3 | | | | | | | |
| 225 | 5.5 | | | | | | | |
| 44' | | | | | | | | |
| 22'46 | | | | | | | | |
| 245 | 1.4 | | | | | | | |
| 2256' | 0.5 | | | | | | | |
| 3'46 | | | | | | | | |
| 22'55' | | | | | | | | |
| 22'3'5' | | | | | | | | |
| 22'446 | 0.2 | | | | | | | |
| 22'33' | | | | | | | | |
| 23'4'5 | 14.2 | | | | | | | |
| 22'455' | 11.8 | | | | | | | |
| 23456 | | | | | | | | |
| 22'33'66' | 17.3 | | | | | | | |
| 33'44' | | | | | | | | |
| 22'44'55' | | | | | | | | |
| 22'344'5 | 0.2 | | | | | | | |
| 22'3455'6 | | | | | | | | |
| 22'33'44' | | | | | | | | |
| 233'44'5 | | | | | | | | |
| Total | 76.0 | | | | | | | |

Sample no. P80-104

Location ST LAWRENCE
STAND BY TRAIN.
AFTER SM. NO SAND.

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 22.1 | | | | | | | |
| 4 | | | | | | | | |
| [illegible] | 11.7 | | | | | | | |
| 22' | | | | | | | | |
| 25 | 2.6 | | | | | | | |
| 23 | 0.6 | | | | | | | |
| 246 | 1.4 | | | | | | | |
| 225 | 0.4 | | | | | | | |
| 44' | | | | | | | | |
| 2246 | 1.7 | | | | | | | |
| 245 | 2.7 | | | | | | | |
| 2256' | | | | | | | | |
| 2346 | | | | | | | | |
| 2'55' | 0.3 | | | | | | | |
| 2235' | | | | | | | | |
| 22446 | | | | | | | | |
| 2255' | | | | | | | | |
| 2345 | 0.4 | | | | | | | |
| 2245 | 1.3 | | | | | | | |
| 23456 | | | | | | | | |
| 2255'6 | | | | | | | | |
| 3344' | | | | | | | | |
| 2244'55' | | | | | | | | |
| 2234'5 | 1.1 | | | | | | | |
| 2234556 | | | | | | | | |
| 2233'44' | | | | | | | | |
| 2334'45 | | | | | | | | |
| Total | 46.9 | | | | | | | |

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPR ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: .....
Source: ..... Region : .....
Program: .....
Date sampled: ..... by: .....
Date analysed: ..... Date reported: .....

Report to: V Oczkiewicz
Address: .....

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | √CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| Pp 3512 | | Train #6 Blank ~ 5 Impingers. | | |
| Pp 3513 | | Kiln A Train 3 Extraction of 5 impingers. | | |
| Pp 3514 | | Clinker Cool. - C Train #7 ~ 3 impingers. | | |
| Pp 3515 | | Kiln B ~ Extraction of 5 impingers | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | PCB Train No. | | | | | | | | | |
| [illegible]12 | | 1614 | | | 2.5 | [illegible] | | | | | |
| [illegible]13 | | 1105 | | | [illegible] | [illegible] | | | | | |
| [illegible]14 | | 1615 | | | | [illegible] | | | | | |
| [illegible]15 | | 1555 | | | | [illegible] | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |

MOE 0051

Sample no. P-3512

Location ST. LAWRENCE CEMENT PLANT
BLANK TRAIN 5 IMPINGERS.

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 4 | | | | | | | | |
| 26 | | | | | | | | |
| 22' | 5408 * | | | | | | | |
| 25 | 41.7 | | | | | | | |
| 23 | 6.7 | | | | | | | |
| 246 | | | | | | | | |
| 225 | 11.1 | | | | | | | |
| 44' | | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 45.4 | | | | | | | |
| 2256' | 29.5 | | | | | | | |
| 2346 | | | | | | | | |
| 22'55' | 37.5 | | | | | | | |
| 22'35' | 51.1 | | | | | | | |
| 22'446 | 21.0 | | | | | | | |
| 22'33' | 35.7 | | | | | | | |
| 23'45 | 61.5 | | | | | | | |
| 22'455' | 10.2 | | | | | | | |
| 23456 | 0.5 | | | | | | | |
| 22'33'66' | 10.7 | | | | | | | |
| 33'44' | 19.3 | | | | | | | |
| 22'44'55' | 0.2 | | | | | | | |
| 22'344'5 | 3.3 | | | | | | | |
| 22'3455'6 | | | | | | | | |
| 22'33'44' | 0.1 | | | | | | | |
| 233'44'5 | 24.4 | | | | | | | |
| Total | 5313 | | | | | | | |

* SUSPECT CONTAMINANT

Sample no. P33-351B

Location ST. LAWRENCE CEMENT PLANT
KILN "A" TRAIN 2 BEFORE [illegible]

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| ? | | | | | | | | |
| 4 | 19.7 | | | | | | | |
| 26 | 63.6 | | | | | | | |
| 22' | 3110.2 | | | | | | | |
| 25 | 288.9 | | | | | | | |
| 23 | | | | | | | | |
| 246 | 35.8 | | | | | | | |
| 225 | 4.3 | | | | | | | |
| 44' | 1076.3 | | | | | | | |
| 2246 | 193.4 | | | | | | | |
| 245 | | | | | | | | |
| 2256' | 9.9 | | | | | | | |
| 2346 | 34.0 | | | | | | | |
| 2255' | 44.8 | | | | | | | |
| 2235' | 1.5 | | | | | | | |
| 22446 | 117.1 | | | | | | | |
| 2233' | 79.8 | | | | | | | |
| 2345 | 72.0 | | | | | | | |
| 22455' | 34.0 | | | | | | | |
| 23456 | 25.6 | | | | | | | |
| 22336'6' | 68.6 | | | | | | | |
| 33'44' | 1607 | | | | | | | |
| 22'44'55' | 156.2 | | | | | | | |
| 22'344'5 | 96.5 | | | | | | | |
| 22'3455'6 | 11.9 | | | | | | | |
| 22'33'44' | 56.2 | | | | | | | |
| 233'44'5 | 40.5 | | | | | | | |
| Total | 5934 | | | | | | | |

Sample no. P-q-351L

Location ST LAWRENCE CEMENT PLANT
CLINKER COOLER TRAIN #17
3 IMPINGERS

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| | 121.3 | | | | | | | |
| 26 | 1.3 | | | | | | | |
| 22' | 9.7 | | | | | | | |
| 25 | | | | | | | | |
| 23 | | | | | | | | |
| 246 | 1.8 | | | | | | | |
| 225 | | | | | | | | |
| 44' | 132.8 | | | | | | | |
| 2246 | 16.0 | | | | | | | |
| 245 | 0.74 | | | | | | | |
| 2256' | 0.17 | | | | | | | |
| 346 | 0.24 | | | | | | | |
| 2255' | 0.47 | | | | | | | |
| 2235' | 7.5 | | | | | | | |
| 2246 | 37.1 | | | | | | | |
| 2235' | 9.7 | | | | | | | |
| 2345 | 35 | | | | | | | |
| 2245' | 0.21 | | | | | | | |
| 23456 | 0.21 | | | | | | | |
| 2233'66' | 9.66 | | | | | | | |
| 33'44' | 1.30 | | | | | | | |
| 2244'5' | 0.97 | | | | | | | |
| 2344'5 | | | | | | | | |
| 23455'6 | | | | | | | | |
| 2233'44' | 12.5 | | | | | | | |
| 2334'45 | 57.9 | | | | | | | |
| Total | 409.8 | | | | | | | |

Sample no. PCB-3515

Location ST LAWRENCE CEMENT PLANT

KILN B EXHAUST E PRECIPITATOR

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 4 | 13.2 | | | | | | | |
| 26 | 59.6 | | | | | | | |
| 22' | 987.3 | | | | | | | |
| 25 | 131 | | | | | | | |
| 23 | 99.7 | | | | | | | |
| 246 | 9.4 | | | | | | | |
| 225 | 49.8 | | | | | | | |
| 44' | 897.7 | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 106.7 | | | | | | | |
| 2256' | | | | | | | | |
| 2346 | 31.3 | | | | | | | |
| 22'55' | 149.6 | | | | | | | |
| 22'3'5' | 164.3 | | | | | | | |
| 22'446 | 34.3 | | | | | | | |
| 22'33' | 97.7 | | | | | | | |
| 23'4'5 | 161.3 | | | | | | | |
| 22'455' | 141.6 | | | | | | | |
| 23456 | 18.0 | | | | | | | |
| 22'33'66' | 276.4 | | | | | | | |
| 33'44' | 17.5 | | | | | | | |
| 22'44'55' | 117.9 | | | | | | | |
| 22'344'5 | 69.3 | | | | | | | |
| 22'3455'6 | 8.9 | | | | | | | |
| 22'33'44' | 80.3 | | | | | | | |
| 233'44'5 | 121.0 | | | | | | | |
| Total | 3907 | | | | | | | |

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

MINISTRY OF THE ENVIRONMENT
LABORATORIES

RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: .....
Source: ..... Region: .....
Program: .....
Date sampled Oct. 31. 1979 by: .....
Date analysed: ..... Date reported: .....

Report to: FRED SEYMOUR
Address: NOCRO TECHNICS

C
Bi
Ba

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| 77-3379 | | Composited Pelletizer Silo 1750 gm | | |
| 77-3380 | | Soxhlet Extraction from Coal | | |
| 77-3381 | | Composited Dust Sample 1720 gm | | |
| 77-3382 | | Kiln #1 Clinker daily Blend from soxhlet extraction | | |
| 77-3383 | | Gravel Bed Sox Extraction " | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
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| | | | | | | | | | | | |

MOE 0 51

Sample no. P.p.-3799

Location [illegible] ST LAWRENCE
PETROCHEMICAL PLANT

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 4 | 32.5 | | | | | | | |
| 26 | 1.8 | | | | | | | |
| 22' | 10.5 | | | | | | | |
| 25 | 5.4 | | | | | | | |
| 23 | 2.7 | | | | | | | |
| 246 | | | | | | | | |
| 225 | 4.0 | | | | | | | |
| 44' | 2.3 | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 10.7 | | | | | | | |
| 2256' | 6.4 | | | | | | | |
| 2346 | | | | | | | | |
| 2255' | 4.5 | | | | | | | |
| 2235' | 1.3 | | | | | | | |
| 2246 | | | | | | | | |
| 2233' | 0.4 | | | | | | | |
| 2345 | 4.7 | | | | | | | |
| 2245' | 1.1 | | | | | | | |
| 23456 | 0.1 | | | | | | | |
| 2233'66' | 1.1 | | | | | | | |
| 33'44' | | | | | | | | |
| 2244'5' | | | | | | | | |
| 2244'5 | | | | | | | | |
| 2345'6 | | | | | | | | |
| 2233'44' | | | | | | | | |
| 2334'5 | | | | | | | | |
| Total | 92.9 | | | | | | | |

Sample no. La - 3360.

Location ST LAWRENCE CEMENT

COAL EXTRACT

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 25.7 | | | | | | | |
| 1 | | | | | | | | |
| 26 | 3.5 | | | | | | | |
| 22' | 49.8 | | | | | | | |
| 25 | 35.4 | | | | | | | |
| 23 | 44.5 | | | | | | | |
| 246 | | | | | | | | |
| 225 | 140.7 | | | | | | | |
| 44' | | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 146.1 | | | | | | | |
| 2256' | 100.2 | | | | | | | |
| 2346 | | | | | | | | |
| 2255' | 161.6 | | | | | | | |
| 2235' | 55.9 | | | | | | | |
| 22446 | | | | | | | | |
| 2253' | 14.3 | | | | | | | |
| 2345 | 64.5 | | | | | | | |
| 22455' | 93.2 | | | | | | | |
| 23456 | 0.1 | | | | | | | |
| 22336'6' | 24.3 | | | | | | | |
| 3344' | 0.3 | | | | | | | |
| 2244'55' | 91.9 | | | | | | | |
| 2234'45 | 9.7 | | | | | | | |
| 22345'56 | | | | | | | | |
| 2233'44' | 0.9 | | | | | | | |
| 2334'45 | 92.4 | | | | | | | |
| Total | 944.7 | | | | | | | |

Samp's no. Pe-3301.

Location ST LAWRENCE

COMPOSITE DUST.

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| | 39.1 | | | | | | | |
| 26 | 2.6 | | | | | | | |
| 22' | 22.4 | | | | | | | |
| 25 | | | | | | | | |
| 23 | 4.1 | | | | | | | |
| 246 | 0.5 | | | | | | | |
| 225 | 5.6 | | | | | | | |
| 44' | 2.2. | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 14.3 | | | | | | | |
| 2256' | 7.6 | | | | | | | |
| 3'46 | | | | | | | | |
| 22'55' | 9.3 | | | | | | | |
| 22'3'5' | 3.0 | | | | | | | |
| 22'446 | | | | | | | | |
| 22'33' | 1.3 | | | | | | | |
| 23'45 | 7.0 | | | | | | | |
| 22'455' | 0.6 | | | | | | | |
| 23456 | | | | | | | | |
| 22'33'66' | 1.5 | | | | | | | |
| 33'44' | 0.7 | | | | | | | |
| 22'44'55' | | | | | | | | |
| 22'344'5 | | | | | | | | |
| 22'3455'6 | | | | | | | | |
| 22'33'44' | 0.2 | | | | | | | |
| 233'44'5 | 4.1 | | | | | | | |
| Total | 126.1 | | | | | | | |

Sample no. Pyc 3502

Location Mile #1 Cuyahoga River

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 7.3 | | | | | | | |
| 4 | | | | | | | | |
| 26 | | | | | | | | |
| 22' | | | | | | | | |
| 25 | | | | | | | | |
| 23 | 4.9 | | | | | | | |
| 246 | | | | | | | | |
| 225 | 6.5 | | | | | | | |
| 44' | 2.0 | | | | | | | |
| 2246 | 1.0 | | | | | | | |
| 245 | 13.0 | | | | | | | |
| 2256' | 7.2 | | | | | | | |
| 2 '46 | | | | | | | | |
| 22'55' | 10.4 | | | | | | | |
| 22'35' | 9.8 | | | | | | | |
| 22'446 | 0.1 | | | | | | | |
| 22'33' | 0.8 | | | | | | | |
| 23'45 | 5.3 | | | | | | | |
| 22455' | 1.5 | | | | | | | |
| 23456 | 0.1 | | | | | | | |
| 22'33'66' | 0.7 | | | | | | | |
| 33'44' | | | | | | | | |
| 22'44'55' | 0.9 | | | | | | | |
| 22'344'5 | | | | | | | | |
| 22'3455'6 | | | | | | | | |
| 22'33'44' | | | | | | | | |
| 233'44'5 | | | | | | | | |
| Total | 64.5 | | | | | | | |

Sample no. P.q-3303

Location St Lawrence cement Plant
GRAVEL BED

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 2.5 | | | | | | | |
| 4 | | | | | | | | |
| 26 | 0.3 | | | | | | | |
| 22' | | | | | | | | |
| 25 | 2.8 | | | | | | | |
| 23 | 2.4 | | | | | | | |
| 246 | 0.2 | | | | | | | |
| 225 | 3.3 | | | | | | | |
| 44' | | | | | | | | |
| 2246 | 1.9 | | | | | | | |
| 245 | 10.7 | | | | | | | |
| 2256' | 6.3 | | | | | | | |
| 246 | | | | | | | | |
| 2255' | 4.9 | | | | | | | |
| 2235' | 2.1. | | | | | | | |
| 22446 | | | | | | | | |
| 2233' | 0.8 | | | | | | | |
| 2345 | 3.5 | | | | | | | |
| 22455' | 0.6 | | | | | | | |
| 23456 | 0.3 | | | | | | | |
| 223366' | | | | | | | | |
| 3344' | | | | | | | | |
| 2244'55' | | | | | | | | |
| 22344'5 | | | | | | | | |
| 223455'6 | | | | | | | | |
| 2233'44' | | | | | | | | |
| 233'44'5 | | | | | | | | |
| Total | 39.6 | | | | | | | |

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: .....
Source: ..... Region :
Program: ..... St Lawrence Cement
Date sampled: ..... by: .....
Date analysed: ..... Date reported: .....

Report to: .....
Address: .....

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| P30-1 | | Final Slurry Sample - 8 hour composite | | |
| | | by Environment Canada Ltd. | | |
| | | - Washing of Precalcined Slurry Feed | | |
| | | 1 - Filter, fine fiber mesh with pore entrainment | | |
| | | 2 - Distillate condensed under silica gel vacuum | | |
| | | 3 - pH not adjusted, with preservative added | | |
| | | prior to analysis | | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| | | | | |

Sample no. P80-1

Location ST. LAWRENCE CEMENT PLANT
FEED SLURRY SAMPLE

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| | | | | | | | | |
| 26 | 12.5 | | | | | | | |
| 22' | | | | | | | | |
| 25 | | | | | | | | |
| 23 | 1.6 | | | | | | | |
| 246 | | | | | | | | |
| 22'5 | | | | | | | | |
| 44' | | | | | | | | |
| 22'46 | | | | | | | | |
| 24'5 | 3.8 | | | | | | | |
| 2256' | | | | | | | | |
| 3'46 | 0.1 | | | | | | | |
| 22'55' | 2.6 | | | | | | | |
| 22'3'5' | 04 | | | | | | | |
| 22'446 | | | | | | | | |
| 22'33' | | | | | | | | |
| 23'4'5 | 0.7 | | | | | | | |
| 22'455' | 2.1 | | | | | | | |
| 23456 | 0.5 | | | | | | | |
| 22'33'66' | 1.1 | | | | | | | |
| 33'44' | 1.1 | | | | | | | |
| 22'44'55' | | | | | | | | |
| 22'344'5 | | | | | | | | |
| 22'3455'6 | | | | | | | | |
| 22'33'44' | | | | | | | | |
| 233'44'5 | | | | | | | | |
| Total | 265 | | | | | | | |

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: ST. LAURENT CEMENT STACK
Source: SAMPLING Region: CRL BURNAL
Program:
Date sampled: JAN 3, 80 by: ENVIROCON
Date analysed: Date reported:
EXTRACTED JUNE 6, 80 BY NTL

Report to:
Address:

C
Bi
Ba

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| P80-83 | | Pentane Blank | | |
| | | - Cotton distilled in glass, dirt for 1 hr | | |
| | | till dry | | |

| LAB. NUMBER | | TOTAL mg Pc.B. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| P80-83 | | | | | | | | | | | |

MOE 0951

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: ST. LAURENT CEMENT STACK
Source: SAMPLING Region: SRL BURN #2
Program:
Date sampled: JAN 3, 80 by: ENVIRACON
Date analysed: Date reported:
EXTRACTED BY JAN 6, 80 BY NTL

Report to:
Address:

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| Page 1 of 2 | #2 | - A stack sample - Impingers (5) contained | | |
| | | containing glassware pieces | | |
| Page 2 of 2 | | - hydrochloric acid + liquid liquid extraction | | |
| | | in each impinger #1-5. | | |
| | | - Samples delivered thru an hydrochloric | | |
| | | acid/distilled water/ethyl ether. | | |
| | | (#1) (2) bottles | | |

| LAB. NUMBER | 1 of 2 2 of 2 | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | |
| Page 2 of 2 | | | | | | | | | | | |
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MOE 0551

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: [illegible]
Source: [illegible] Region: [illegible]
Program:
Date sampled: [illegible] by: [illegible]
Date analysed: [illegible] Date reported: [illegible]

Report to:
Address:

| | |
|---|---|
| C | |
| Bi | |
| Ba | |

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| [illegible] | [illegible] | Train — not a stock sample — Partial Contd. | | |
| | | — Subject retest after Blank which the | | |
| [illegible] | | Coastal Extraction Enzymatic proteins + Bot Extract | | |
| | | + Bat Bait + Rose Lemon as + samples listed | | |
| | | — Continue Recipe with Bottle Number 2 | | |
| | | — Media Contact Treatment # Test 2 | | |
| | | | | |
| | | | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | |
| [illegible] | | | | | | | | | | | |
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MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: [illegible]
Source: [illegible] Region [illegible]
Program: [illegible]
Date sampled: [illegible] by: [illegible]
Date analysed: [illegible] Date reported: [illegible]

Report to: [illegible]
Address: [illegible]

| | |
|---|---|
| C | |
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| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | ✓CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| [illegible] | [illegible] | [illegible] | | |
| | | [illegible] | | |
| [illegible] | | [illegible] | | |
| | | [illegible] | | |
| | | [illegible] | | |
| | | [illegible] | | |
| | | [illegible] | | |
| | | [illegible] | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| [illegible] | | | | | | | | | | | |
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MOE 0-65

MAIL ADDRESS
MINISTRY OF THE ENVIRONMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

# MINISTRY OF THE ENVIRONMENT
## LABORATORIES

EXPRESS ADDRESS:
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality: [illegible] Report to: [illegible]
Source: [illegible] Region Address: [illegible]
Program: [illegible]
Date sampled: [illegible] by: Environ.
Date analysed: [illegible] Date reported: [illegible]
[illegible] see [illegible] note.

C
Bi
Ba

| LAB. NUMBER | SENDER'S NUMBER | SAMPLING POINT LOCATIONS AND TIME | NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC. | √CHECK BELOW IF CHLORINE PRESENT |
|---|---|---|---|---|
| From | 3 | - Kstrak sample, Florisil cartridge | | |
| | | Soxtec Extraction Pentane 2 hrs. & 45 min. & 150 C | | |
| | | set of 3 one hour & rinsed acetone 2x 2 min. with | | |
| [illegible] | | - Contains Pravachol Mat Bath Bottles, Lan [illegible] | | |
| | | - Florisil cartridge Prepared by | | |

| LAB. NUMBER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| [illegible] | [illegible] | | | | | | | | | | |

MOE 0651

Sample no. P80-89

Location ST LAWRENCE
PENTAGE PLUS

| PCB isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 4 | | | | | | | | |
| 26 | 16.1 | | | | | | | |
| 22' | | | | | | | | |
| 25 | 0.6 | | | | | | | |
| 23 | 0.5 | | | | | | | |
| 246 | | | | | | | | |
| 225 | | | | | | | | |
| 44' | | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 1.3 | | | | | | | |
| 2256' | | | | | | | | |
| 2346 | 0.2 | | | | | | | |
| 22'55' | | | | | | | | |
| 22'3'5' | | | | | | | | |
| 22'446 | | | | | | | | |
| 22'33' | | | | | | | | |
| 23'4'5 | | | | | | | | |
| 22'455' | 0.2 | | | | | | | |
| 23456 | | | | | | | | |
| 22'33'66' | 0.4 | | | | | | | |
| 33'44' | 3.0 | | | | | | | |
| 22'44'55' | | | | | | | | |
| 22'344'5 | 17.5 | | | | | | | |
| 22'3455'6 | | | | | | | | |
| 22'33'44' | | | | | | | | |
| 233'44'5 | | | | | | | | |
| Total | 39.8 | | | | | | | |

Sample no. P20-90

Location [illegible] ST [illegible]
[illegible] & R.
& STARK. [illegible]

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 4 | 1187.5 | | | | | | | |
| 26 | 149.6 | | | | | | | |
| 22' | | | | | | | | |
| 25 | 222.9 | | | | | | | |
| 23 | 203.7 | | | | | | | |
| 246 | | | | | | | | |
| 225 | 99.6 | | | | | | | |
| 44' | | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 394.2 | | | | | | | |
| 2256' | 392.5 | | | | | | | |
| 2346 | | | | | | | | |
| 2255' | 642.9 | | | | | | | |
| 2235' | 194.0 | | | | | | | |
| 22446 | | | | | | | | |
| 2233' | 140.3 | | | | | | | |
| 2345 | 312.0 | | | | | | | |
| 22455' | 213.0 | | | | | | | |
| 23456 | | | | | | | | |
| 22336' | 207.6 | | | | | | | |
| 33'44' | 10.4 | | | | | | | |
| 22'44'55' | 75.2 | | | | | | | |
| 22'3445 | 17.4 | | | | | | | |
| 22'3455'6 | 2.5 | | | | | | | |
| 22'33'44' | 26.2 | | | | | | | |
| 233'44'5 | 16.2 | | | | | | | |
| Total | ~~4519.7~~ | | | | | | | |

3325

Samp's no. P80-91

Location ST LAWRENCE

STANDBY STA. NO. P1 2/1/81

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 4 | | | | | | | | |
| 26 | | | | | | | | |
| 22' | | | | | | | | |
| 25 | | | | | | | | |
| 23 | 4.8 | | | | | | | |
| 246 | 0.5 | | | | | | | |
| 225 | 5.3 | | | | | | | |
| 44' | | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 8.5 | | | | | | | |
| 2256' | 0.3 | | | | | | | |
| 2346 | 1.9 | | | | | | | |
| 2255' | 5.8 | | | | | | | |
| 2235' | | | | | | | | |
| 22446 | 10 | | | | | | | |
| 2233' | 0.3 | | | | | | | |
| 2345 | 0.5 | | | | | | | |
| 22455' | 0.4 | | | | | | | |
| 23456 | 0.1 | | | | | | | |
| 223366' | | | | | | | | |
| 3344' | | | | | | | | |
| 2244'55' | | | | | | | | |
| 23445 | | | | | | | | |
| 23455'6 | | | | | | | | |
| 2233'44' | | | | | | | | |
| 2334'45 | 36.3* | | | | | | | |
| Total | ~~65.7~~ 29.4 | | | | | | | |

Samp'e no. PBO-92

Location ST LAWRENCE.
A STACK. TRAIN #1.
FLORISIL

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | | | | | | | | |
| 4 | | | | | | | | |
| 26 | 102.1 | | | | | | | |
| 22' | 576.0 | | | | | | | |
| 25 | 749.1 | | | | | | | |
| 23 | 464.3 | | | | | | | |
| 246 | | | | | | | | |
| 225 | 851.4 | | | | | | | |
| 44' | | | | | | | | |
| 2246 | | | | | | | | |
| 245 | 629.7 | | | | | | | |
| 2256' | 471.7 | | | | | | | |
| 246 | | | | | | | | |
| 22'55' | 714.7 | | | | | | | |
| 22'3'5' | 295.5 | | | | | | | |
| 22'446 | | | | | | | | |
| 22'33' | 74.6 | | | | | | | |
| 23'45 | 259.5 | | | | | | | |
| 22'455' | 117.5 | | | | | | | |
| 23456 | | | | | | | | |
| 22'33'66' | 114.1 | | | | | | | |
| 33'44' | 6.2 | | | | | | | |
| 22'44'55' | 45.1 | | | | | | | |
| 22'344'5 | 22.1 | | | | | | | |
| 22'3455'6 | | | | | | | | |
| 22'33'44' | | | | | | | | |
| 233'44'5 | | | | | | | | |
| Total | 5493.6 | | | | | | | |

Sample no. P80-93

Location ST LAWRENCE R. STACK TRAIN #3.
FLORAL

| PCB Isomer | ng. Detected | Total Chlorobiphenyl | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Mono- | Di- | Tri- | Tetra- | Penta- | Hexa- | Hepta- |
| 2 | 1669.9 | | | | | | | |
| 4 | 2961.8 | | | | | | | |
| .26 | 116.6 | | | | | | | |
| 22' | 623.3 | | | | | | | |
| 25 | 1441.6 | | | | | | | |
| 23 | 1240.9 | | | | | | | |
| 246 | | | | | | | | |
| 22'5 | 727.9 | | | | | | | |
| 44' | | | | | | | | |
| 22'46 | | | | | | | | |
| 245 | 890.0 | | | | | | | |
| 2256' | 711.2 | | | | | | | |
| 2 46 | | | | | | | | |
| 22'55' | 806.7 | | | | | | | |
| 22'3'5' | 256.1 | | | | | | | |
| 22'446 | | | | | | | | |
| 22'33' | 86.6 | | | | | | | |
| 23'4'5 | 280.6 | | | | | | | |
| 22'455' | 137.9 | | | | | | | |
| 23456 | | | | | | | | |
| 22'33'66' | 140.6 | | | | | | | |
| 33'44' | 2.9 | | | | | | | |
| 22'44'55' | 49.5 | | | | | | | |
| 22'344'5 | 39.5 | | | | | | | |
| 22'3455'6 | 34.1 | | | | | | | |
| 22'33'44' | 25.7 | | | | | | | |
| 233'44'5 | 4.4 | | | | | | | |
| Total | 12,247.8 | | | | | | | |

# APPENDIX 4

# Conditions for Gas Chromatography with Capillary Column

Gas chromatographic conditions:

Chromatograph: H & P 5840A

Column: Fused Silica, coated with SP-2100, 50 m long, I.D. 0.2mm

Carrier Gas: Helium, linear velocity 23 cm/min at 130°C

Detector: ECD with 25 ml $N_2$/min as make-up gas

Column Temperature: 70°C for 1 min, 20°C/min to 130°C, then 2°C/min to 250°C, hold at 250°C for 20 min.

Injection: splitless with iso-octane, 1 ul

Calibration with the following PCB's: 2-; 3-; 4-; 2,2'-; 2,4-; 2,3-; 3,5-; 2,4,6-; 3,3'-; 3,4-; 2,2'5-; 4,4'-; 2,2',6,6'-; 2,4,5-; 2,3',5-; 2,4',5-; 2,3'4'-; 2,2',5,5'-; 2,2',4',5-; 2,2',4,4'-; 2,3,5,6-; 2,2',4,6,6'-; 2,2',3',5-; 2,3',5,5'-; 2,2',3,3'-; 2,2',4,5',6-; 2,2',4,4',6-; 2,3,4,5-; 2,3',4',5-; 2,3',4,5',6-; 2,2',4,4',6,6'-; 2,2',4,5,5'-; 2,3',4,4',6-; 2,2',3,4,5'-; 3,3',4,4'-; 2,2',4,4',5',6-; 2,2',3,5,5',6-; 2,2',3,4,4',6-; 2,2',3,4,5,6'-; 2,2',4,4',5,5'-; 2,2',3,4,5,5'-; 2,2',3,4,4',5-; 2,2',3,3',4,5-; 2,2',3,3',4,4'-; 2,3,3',4,4',5-

Data acquisition and calculation: with the build in data acquisition system and integrator. As PCB's were identified only the peaks, which were within ± 0.25% of the retention time of the standards.

Discussion

The gas chromatographs indicate, that the extracts from coal, filter-coke, impingers and "Florisil" cartridges contain a very complicated mixture of organic compounds. Only peaks with retention times within ± 0.25% of the used standards were identified as PCB's. However, because of the complicated nature of the samples, there exists the possibility that some peaks identified as PCB's are in reality interfering compounds or mixture of PCB and interfering compound. On the other hand, some peaks which were not identified as PCB's, because standards were not available might well be PCB's.

The reported values are the best values as they can be reported, when using the commercials available PCB's standards and high resolution gas chromatography with capillary column.

## Ammounts of PCBs Spiked on Stack Samples

| | November 2, 1979 | January 3, 1980 |
|---|---|---|
| | Train A | Train 2 |
| Filter | 89.05 ug | 188.2 ug |
| Impinger | 89.05 ug | 188.2 ug |
| Florisil | 267.15 ug | 564 ug |